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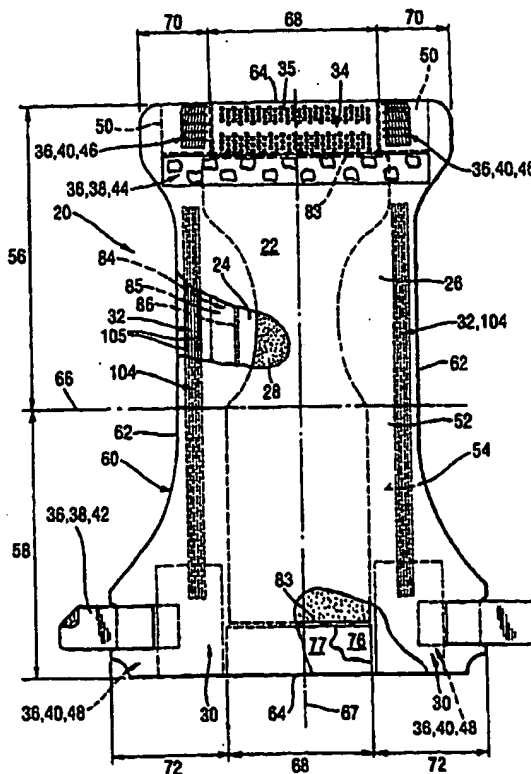
## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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(54) Title: LIQUID DISTRIBUTION MATERIALS WITH IMPROVED DISTRIBUTION PROPERTIES UNDER SUB-SATURATION

## (57) Abstract

The present invention is a fluid handling member having improved balance of permeability, ability to release fluid both at full and partial saturation. This member has (i) a sufficiently open structure with a permeability of at least about 1 Darcy at 100 % saturation, (ii) the ability to readily release the fluid contained therein by having a low Capillary Sorption Desorption Height (CSDH 50) of less than about 150 cm, and (iii) a permeability at 50 % of their saturation, which is more than about 14 % of the permeability at 100 % saturation. A preferred execution of such materials comprises an open celled foam material, such as of a High Internal Phase Emulsion (HIPE) type. Such materials are particularly useful for absorbent articles, such as diapers, adult incontinence articles, or feminine hygiene articles.



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## LIQUID DISTRIBUTION MATERIALS WITH IMPROVED DISTRIBUTION PROPERTIES UNDER SUB-SATURATION

### General field of the invention

The present invention relates to fluid handling members exhibiting improved liquid handling properties, which make them particularly useful in hygienic absorbent articles such as baby diapers, training pants, adult incontinence articles, feminine hygiene articles, which can be used for receiving with liquid bodily discharges such as urine, menses, sweat, or low viscosity fecal matter.

### Background / Prior art

In the general field of disposable absorbent articles and structures, materials exhibiting specific fluid distribution properties are well known. Such materials became more and more relevant with the introduction of highly absorbent materials, also called Absorbent Gelling Materials or superabsorbent materials or shortly superabsorber, which do provide a good means for storing aqueous fluids such as urine, but do not enhance fluid transport, and even reduction of fluid transport can occur, when sub-optimal designs and/or suboptimal materials are employed, and phenomena often referred to as "gel-blocking" take place. For example, in structures where the superabsorbent is homogeneously mixed with cellulose fibers, a certain critical concentration, which is strongly depending on the choice of the superabsorbent material, should not be exceeded in order to not deteriorate efficacy of the absorbent core.

As a consequence, a vast number of absorbent core designs have appeared with a separated functionality, such as by comprising not only liquid storage regions or materials, but also regions with specialized properties for enhanced acquisition and/or distribution of the fluid. Often, one region aimed at enhancing acquisition and distribution at the same time.

Initially, the requirements for a distribution material were not very high, and standard paper tissue materials such as used as wrapsheets in the cores and described for example in US-A-3.952.745 (Duncan), were applied to also enhance the fluid distribution, as described in EP-0 343 941 (Reising) or US-A-4.578.068 (Kramer).

Further developments can be exemplified by EP-A- 0.397.110 (Latimer) disclosing an absorbent article comprising a surge management portion for improved fluid handling, having specific basis weights, acquisition times and residual wetness; US-A-4.898.642 (Moore et al.) discloses specially twisted, chemically stiffened cellulosic fibers and absorbent structures made therefrom; EP-A-0.640.330 (Bewick-Sonntag et al.) discloses the use of such fibers in a specific arrangement with specific superabsorbent materials.

Further approaches aimed at improving the wicking properties of cellulose fiber based materials, such as US-A-3.575.174 or US-A-4.781.710, whereby parts of the structure are compressed to a higher density, thus creating smaller pores for increased wicking height for example along "wicking lines" or in closed mesh patterns.

As some of these materials did exhibit an undesired hard feel, methods for post formation treatments were well known to improve softness. "Post formation treatment" refers to the fact that - instead of or in addition to increasing softness during the making or formation of the tissue - the tissue is treated mechanically in a separate process step after forming and drying of the tissue, often just prior to further processing such as combining the tissue with other materials to form an absorbent core or article. Examples for such treatments are US-A-5.117.540 (Walton) or US-A-4.440.597 (Wells).

Other attempts to impact on the pore size of distribution materials is described in US-A-5.244.482 (Hassenboehler), aiming at reducing maximum pore size by stretching a fibrous structure comprising meltable fibers in one direction and "freezing" the deformation by heat curing.

Also, special material composites were developed, aiming at a allowing to tailor the pore size and pore size distribution. Examples for such improvements are described in greater detail in US-A-5.549.589 (Horney et al.) or in PCT application WO 97/38654 (Seger et al.). Both aim essentially at providing a resilient structure by using specially stiffened cellulosic fibers such as crosslinked cellulose soft-wood fibers, and by filling the large pores with small and thin cellulosic fibers such as eucalyptus fibers. Both applications further add means for providing sufficient integrity and strength to the structure, the first one (US-A-

5.549.589) by adding thermoplastic fibers and partially melt these, the second (WO 97/38654) by adding a chemical binder.

A further approach as disclosed in EP Application EP-A-0.810.078 (d'Acchioli et al.) uses a special post-formation mechanical treatment of webs, thereby imparting improved fluid handling properties such as described by higher liquid flux rates at certain wicking heights.

With the wish to improve the functionality of the absorbent articles, more specific requirements for distribution materials developed, such that porous materials were investigated in more depth. In order to improve the longitudinal fluid distribution, high surface area synthetic fibers were applied in absorbent structures, such as described in US Statuary Invention Registration H1511. Another class of materials are foamed structures, such as cellulosic foams such as commercially available by Spontex SA. France.

Other polymeric foams for being used in absorbent articles were disclosed in US-A-5.268.224 (DesMarais), namely High Internal Phase polymerized materials, which can be used for storing liquids, and have at the same time the ability to avoid localized saturation, by spreading the stored fluid throughout the material.

However, all these investigations so far aimed at improving the wicking properties of the distribution materials such as flux, wicking height and wicking times, but failed to recognize the importance of the dewatering mechanism of the distribution materials by the liquid storage materials, especially when such materials are not fully saturated, such as can be relevant in absorbent articles between multiple loadings. Cofiled Patent Application (attorney docket number CM1698) has recognized the importance of providing storage cores with high capillary suction to dewater the distribution materials. This approach, however, is critically hinging on the suction properties of the storage material.

#### Objects of the invention

Henceforth, it is an object of the present invention to provide materials which can be readily dewatered , especially under low saturation conditions.

It is another object of the present invention, to provide materials which transport liquid more readily even at a low or moderate degree of saturation.

It is a further object of the present invention to construct absorbent articles comprising such distribution members, thereby having an improved liquid handling performance.

#### Summary

The present invention relates to fluid handling members, with improved balance of permeability, ability to release fluid both at full and partial saturation.

Specifically, these materials have a sufficiently open structure by having a permeability at 100% saturation,  $k(100)$ , which has a value of more than about 1 Darcy, preferably more than about 2 Darcy, even more preferably more than 8 Darcy or even more than 100 Darcy.

Further, these materials readily release the fluid contained therein, by having a Capillary Sorption Desorption Height at 50% of the capacity at 0 cm height (CSDH 50) value of less than about 150 cm, preferably less than about 100 cm, even more preferably less than 75 cm, or even less than 50 cm.

Even further, these materials have a permeability at 50% of their saturation,  $k(50)$ , which is more than about 14% of the permeability at 100% saturation,  $k(100)$ , preferably more than about 18%, even more preferably more than about 25% or even more than about 35%.

Preferred materials have a permeability at 30% of their saturation,  $k(30)$ , which is more than about 3.5% of the permeability at saturation, even more preferably more than about 5%, or even more than 10%.

A preferred execution of such materials comprises an open celled foam material, such as of a High Internal Phase Emulsion (HIPE) type.

Such materials are particularly useful for absorbent articles, such as diapers, adult incontinence articles, or feminine hygiene articles.

#### Brief description of drawings

Figures 1 to 4 show different executions of the permeability test set up. Figures 1 and 2 relate to a simplified test. Figures 3 and 4 relate to a general test. Figures

1 and 3 relate to the measurement of the transplanar permeability, and Figures 2 and 4 to the in-plane permeability.

Fig.5 - Shows the Capillary Sorption test stand (Capsorption)

Fig.6 - Shows a diaper as example for an absorbent article

Fig.7 - Shows the Vertical Wicking Flux test stand

#### Detailed description

As used herein, the term "fluid handling member" refers to the components of the absorbent article that typically provide at least the fluid handling functionality. An absorbent article can comprise one or more of the various fluid handling members, such as one or more fluid acquisition member, one or more fluid distribution members and / or one or more fluid storage members. Each of these members can comprise one or more sub-elements, which can be homogeneous or not, i.e. each member can be made from one material or from several materials. For example, such members can be layers, optionally consisting of sub-layers, and or optionally having different composition, or density, or thickness.

Each of these members can have a specialized functionality, such primarily providing acquisition functionality or primarily providing fluid storage functionality. Alternatively, members can have multiple functionality, such as the very first "cellulose only" diapers wherein the cellulose fluff performed acquisition, distribution and ultimate storage functionality at the same time.

The "storage absorbent member" refers to the absorbent member(s) of the absorbent core that function primarily to ultimately store absorbed fluids.

A "fluid distribution member" in the meaning of the present invention is a member, which satisfies the requirements as laid out for the fluid distribution functionality, regardless whether the member also has some other fluid handling functionality.

A "fluid acquisition member" refers to parts of the absorbent core, which are primarily designed to receive the liquid as it reaches the absorbent article.

As used herein, the term "absorbent core" refers to the members of the absorbent article that are primarily responsible for fluid handling of the article, thus including the "fluid handling member(s)". As such, the absorbent core



typically does not include the topsheet or backsheet of the absorbent article, though in certain instances the topsheet could be considered, for example, to provide specific fluid acquisition performance.

An absorbent core can be divided into "regions" of the core, wherein such "regions" can perform the functionality of one or more of the members as outlined above. Thus, an acquisition region can comprise an acquisition member (and also comprise other members), it can consist of an acquisition member (and nothing else), which can consist of an acquisition material. Or, an acquisition/distribution region can comprise both an acquisition member and an distribution member.

As used herein, the term "absorbent articles" refers to devices which absorb and contain body exudates, and, more specifically, refers to devices which are placed against or in proximity to the body of the wearer to absorb and contain the various exudates discharged from the body. As used herein, the term "body fluids" includes but is not limited to urine, menses, vaginal discharges, sweat and feces.

The term "disposable" is used herein to describe absorbent articles which are not intended to be laundered or otherwise restored or reused as an absorbent article (i.e., they are intended to be discarded after use and, preferably, to be recycled, composted or otherwise disposed of in an environmentally compatible manner).

As used herein, the term "Z-dimension" refers to the dimension orthogonal to the length and width of the member, core or article. The Z-dimension usually corresponds to the thickness of the member, core or article. As used herein, the term "X-Y dimension" refers to the plane orthogonal to the thickness of the member, core or article. The X-Y dimension usually corresponds to the length and width, respectively, of the member, core or article.

As used herein, the terms "region(s)" or "zone(s)" refer to portions or sections of the absorbent member. Thereby, the region(s) or zone(s) can be two-dimensional (front / back) or can be three-dimensional (like an acquisition region having - even if it were in the form of a layer - a three-dimensional extension).

As use herein, the term "layer" refers to an absorbent member whose primary dimension is X-Y, i.e., along its length and width. It should be understood that the term layer is not necessarily limited to single layers or sheets of material. Thus the layer can comprise laminates or combinations of several sheets or webs of the requisite type of materials. Accordingly, the term "layer" includes the terms "layers" and "layered".

For purposes of this invention, the term "upper" should be understood to refers to absorbent members, such as layers, that are nearest to the wearer of the absorbent article, and typically face the topsheet of an absorbent article; conversely, the term "lower" refers to absorbent members that are furthestmost away from the wearer of the absorbent article and typically face the backsheet.

All percentages, ratios and proportions used herein are calculated by weight unless otherwise specified.

#### Improved distribution member

The requirements for the distribution members can be determined by either looking at the member or at the materials contained in said member. Henceforth, the requirements as laid out in the present description have to be satisfied for either the total member or for the respective materials therein.

Henceforth, distribution members or materials useful for such members according to the present invention can be described by the following important parameter:

First, the permeability at full saturation ( $k_{100}$ ) of the member or material. Conventional distribution materials have this permeability balanced so as to find the optimum between having little resistance to the fluid flow (i.e. high permeability) and sufficient capillary pressure so as to provide wicking properties, such as results from smaller pore sizes (i.e. lower permeability). The permeability at full saturation ( $k_{100}$ ) should generally be more than 1 Darcy (with 1 Darcy corresponding to  $9.869 \times 10^{-13} \text{ m}^2$ ), preferably more than 2 Darcy, or even 8 Darcy, or even more preferably more than 100 Darcy. The full saturation can be determined by the Capsorption test as described

hereinafter as the maximum uptake, corresponding to the result Capillary Sorption Absorption Capacity at 0 cm height (CSAC 0)

Second, the dependency of the permeability on the degree of saturation. This property has not been considered in previous material design considerations, and conventional materials have a strongly sub-proportional behavior, i.e. the actual permeability at a degree of saturation of less than 100% is significantly lower than it would be for a linear correlation between actual permeability and saturation.

Thirdly, the Capillary Sorption Pressure, namely the Capillary Sorption Desorption pressure, such as measured in the Capillary Sorption Test as described hereinafter. This parameter describes the ability of the materials or members to release liquids, to satisfy their role as a distribution element in an absorbent article.

Additionally and often preferably, the distribution materials may satisfy the requirement of high fluid flux rates in the vertical wicking flux test as described herein after. Preferably, the materials provide at a wicking height of 15 cm a flux of at least  $0.045 \text{ g/cm}^2/\text{sec}$ , preferably more than  $0.06 \text{ g/cm}^2/\text{sec}$ , and even more preferably more than  $0.10 \text{ g/cm}^2/\text{sec}$ .

With careful selection of materials satisfying the right balance of the these parameter, important benefits can be realized for the absorbent structures and/or respective articles.

First, the liquid distribution materials are readily dewatered after they have been loaded such as with a urine gush. This is relevant so as to allow these materials to be ready for receiving a subsequent loading as it often occurs in real use.

Second, these materials allow a more even liquid distribution of liquids, even at loads which are relatively small compared to the design capacity. This is even more important for designs which aim at maintaining an improved fit on the wearer by avoiding high liquid accumulation in certain regions of the article, but rather aim for an even distribution of the liquid stored.

Thirdly, if the materials also satisfy the high flux requirements, the liquid can be well and quickly distributed even against gravity. This becomes particularly relevant, if the ultimate storage of the fluid is intended to be distant to the loading zone or area. These materials are therefore especially useful in core designs such as described in PCT Application US 97/05046 filed on March 27, 1997.

The permeability of the materials or members are determined by the permeability tests as described hereinafter.

Without wishing to be bound by the theory, it is believed, that the actual permeability  $k\{S\}$  has a dependency from the degree of saturation, which for many relevant systems can be approximated by the following equation (see also "Dynamics of fluids in porous media" by J.Bear, Haifa, publ. Dover Publications, Inc., New York, 1988, esp. pages 461ff, 491ff):

$$k\{S\} = k\{100\} * \{S^{SDP}\}$$

wherein  $k$  denotes the permeability in units of Darcy; and SDP represents the dimensionless exponent or Saturation Dependency Parameter describing the sub-proportional behavior.  $S$  denotes the degree of saturation, ranging from 0 to 1, wherein the value of 1 corresponds to full saturation (i.e. 100% saturation) under zero external and/or capillary pressure).

Conventional design criteria for distribution materials focused on high values for permeability at saturation ( $k(100)$ ), which of course could lead to structures having little or no wicking capability, thus being suitable as acquisition material, wherein essentially the "free flow regime" should be controlled, but not for distribution materials. Such materials would have very poor transport properties under wicking conditions, such as transporting against gravity. Such extreme properties are found in conventional acquisition materials, though distribution material such as described in European Patent Application EP-A-0. 809.991 provide a combination of a wicking ability and free flow control - but still under full saturation conditions.

Materials according to the present invention exhibit a permeability  $k(100)$  of at least 1 Darcy, preferably at least 2 Darcy. Higher values for the permeability provide an even less reduced resistance to the fluid transport, and are preferred as long as this is achieved without violating the further requirements as laid out

herein. In particular, materials having a permeability of more than 8 Darcy or even more than 100 Darcy can be very suitable.

As can be seen from the equation, a higher value for the SDP parameter describes systems with a stronger sub-proportional behavior - if the SDP were equal to one, a linear relation would exist. Conventional distribution materials exhibit a strong sub-proportional behavior, such as can be described by SDP having values of 3 or more. For such a value, the permeability at 50% saturation is only 12.5% of the permeability at 100% saturation, thus also the ability for receiving and distributing further liquid load is dramatically reduced.

Henceforth, materials according to the present invention have a SDP value of less than 3, preferably less than 2.75, even more preferably less than 2.5, and values of less than 2 are even better. Such values correspond to a permeability at 50% saturation of more than 14% of the permeability at 100% saturation, preferably more than about 18%, even more preferably more than about 25% , and values of more than 35% are even better. Such values correspond to a permeability at 30% saturation of more than about 3.5% of the permeability at 100% saturation, preferably more than about 5%, even more preferably more than about 10 %.

The simplified permeability test as laid out hereinafter can measure the "trans-planar" permeability, i.e. the permeability in the thickness dimension of the sample as determined, and - with a modified sample cell - also the "in-plane" permeability. For a number of materials, such as isotropically foamed foams, the trans-planar and the in-plane permeability will be essentially identical. This Simplified Permeability Test provides a simple test set up to measure permeability for two special conditions: Either the permeability can be measured for a wide range of porous materials (such as non-wovens made of synthetic fibres, or cellulosic structures) at 100% saturation, or for materials, which reach different degrees of saturation with a proportional change in caliper without being filled with air (respectively the outside vapour phase) for which the permeability at varying degrees of saturation can readily be measured at various thicknesses.

For example the described collapsible foams exhibit a thickness or caliper, which is dependent on the degree of fluid load or saturation, i.e. they have a certain thickness at saturation, which is being reduced upon removal of fluid, as

the foam pores are of such a size, that they collapse upon removal of liquid from them. Conversely, a certain caliper can be set to define a certain degree of loading. Thus, such materials the Simplified Permeability Test can be readily applied to determine the dependency of the permeability on the saturation.

The General Permeability Test as described hereinafter is useful for determining the dependency of the permeability on the saturation for porous materials in the general sense, such as fibrous webs or structures, or foams which maintain their pore size essentially independent of the degree of wetting.

A further important requirement for the materials or members according to the present invention is their ability to release the fluid to a storage medium. This reflects the fact, that the distribution materials or members should not retain the liquid for too long times, but only for the time that is required to transport the fluid to the appropriate storage material of member.

A suitable parameter to describe this property is the Capillary Sorption Desorption pressure, as determined via the member's ability to receive and to release fluid at varying capillary pressures, herein determined in units of water column height ("capillary height"), which are generally encountered when the member is positioned in an absorbent article. The Capillary Sorption Absorbent Capacity test (also referred to herein as the Capsorption test) measures the amount of test fluid per gram of an absorbent member or material that is taken up or released when the material or member is placed at varying heights on a capillary sorption apparatus. The Capillary Sorption Absorbent Capacity test is described in greater detail in the Test Methods section below, yielding the Capillary Sorption Desorption Height at which the material has released 50% of the amount of fluid at 0 cm sorption height (CSDH 50).

Materials useful in the context of the present invention should have a CSDH 50 of less than 150 cm, preferably less than 100cm, even more preferably less than 75 cm or even less than 50 cm.

Materials particularly useful for being used for the present invention are hydrophilic, flexible polymeric foam structures of interconnected open-cells.

For such foams, the mechanical strength of the foam can be such that, upon giving up its liquid, the foam collapses under the capillary pressures involved. The collapse process reduces the effective foam capacity by a substantial factor related to the density of the foam, as is described hereinafter. The collapse, if relatively uniform throughout the structure, also reduces the amount of liquid held in place at the point of liquid insult. In this regard, the strength of the foams is less than the capillary pressure exerted by the foams such that the foams will collapse when the aqueous liquids are removed by the storage component of the core. Capillary pressure is controlled herein primarily by adjusting foam cell size (which relates inversely to surface area per unit volume). Strength is controlled by the combination of crosslink density and foam density, which can be expressed as crosslink density per unit volume as defined hereinafter. The type of crosslinker and other comonomers can also be influential.

Polymeric foams useful herein are those which are relatively open-celled. The cells in such substantially open-celled foam structures have intercellular openings or "windows" that are large enough to permit ready liquid transfer from one cell to the other within the foam structure.

These substantially open-celled foam structures will generally have a reticulated character with the individual cells being defined by a plurality of mutually connected, three dimensionally branched webs. The strands of polymeric material making up these branched webs can be referred to as "struts." For purposes of the present invention, a foam material is "open-celled" if at least 80% of the cells in the foam structure that are at least 1  $\mu\text{m}$  in size are in fluid communication with at least one adjacent cell.

In addition to being open-celled, these polymeric foams are sufficiently hydrophilic to permit the foam to absorb aqueous liquids. The internal surfaces of the foam structures are rendered hydrophilic by residual hydrophilizing surfactants and/or salts left in the foam structure after polymerization, or by selected post-polymerization foam treatment procedures, as described hereafter.

The extent to which these polymeric foams are "hydrophilic" can be quantified by the "adhesion tension" value exhibited when in contact with an absorbable test liquid. The adhesion tension exhibited by these foams can be determined experimentally using a procedure where weight uptake of a test

liquid, e.g., synthetic urine, is measured for a sample of known dimensions and capillary suction specific surface area. Such a procedure is described in greater detail in the Test Methods section of U.S. Patent No. 5,387,207 (Dyer et al.) issued Feb. 7, 1995, which is incorporated by reference. Foams which are useful as distribution materials of the present invention are generally those which exhibit an adhesion tension value of from about 15 to about 65 dynes/cm, more preferably from about 20 to about 65 dynes/cm, as determined by capillary suction uptake of synthetic urine having a surface tension of  $65 \pm 5$  dynes/cm.

An important aspect of these foams is their glass transition temperature ( $T_g$ ). The  $T_g$  represents the midpoint of the transition between the glassy and rubbery states of the polymer. Foams that have a higher  $T_g$  than the temperature of use can be very strong but can also be very rigid and potentially prone to fracture. Such foams also tend to creep under stress and be poorly resilient when used at temperatures colder than the  $T_g$  of the polymer. The desired combination of mechanical properties, specifically strength and resilience, typically necessitates a fairly selective range of monomer types and levels to achieve these desired properties.

For distribution foams useful for the present invention, the  $T_g$  should be as low as possible, so long as the foam has acceptable strength. Accordingly, monomers are selected as much as possible that provide corresponding homopolymers having lower  $T_g$ 's.

The shape of the glass transition region of the polymer can also be important, i.e., whether it is narrow or broad as a function of temperature. This glass transition region shape is particularly relevant where the in-use temperature (usually ambient or body temperature) of the polymer is at or near the  $T_g$ . For example, a broader transition region can mean transition is incomplete at in-use temperatures. Typically, if the transition is incomplete at the in-use temperature, the polymer will evidence greater rigidity and will be less resilient. Conversely, if the transition is completed at the in-use temperature, then the polymer will exhibit faster recovery from compression. Accordingly, it is desirable to control the  $T_g$  and the breadth of the transition region of the polymer to achieve the desired mechanical properties. Generally, it is preferred that the  $T_g$  of the polymer be at least about  $10^\circ\text{C}$  lower than the in-use temperature. (The  $T_g$  and the width of the transition region are derived from the loss tangent



vs. temperature curve from a dynamic mechanical analysis (DMA) measurement, as described in U.S. Patent No. 5,563,179 (Stone et al.) issued Oct. 8, 1996.)

Polymeric foams useful for the present invention can be described by a number of parameter.

Foams useful for the present invention are able to wick aqueous liquids to a significant height against the force of gravity, e.g., at least about 15 cm. The column of liquid held within the foam exerts a significant contractile capillary pressure. At a height determined by both the strength of the foam (in compression) and the surface area per unit volume of the foam, the foam will collapse. This height is the Capillary Collapse Pressure (CCP) expressed in cm at which 50% of the volume of the foam at zero head pressure is lost. Preferred distribution foams useful for the present invention will have a CCP of at least about 15 cm, more preferably at least about 20 cm, still more preferably at least about 25 cm. Typically, preferred distribution foams will have a capillary collapse pressure of from about 15 cm to about 50 cm, more preferably from about 20 cm to about 45 cm, still more preferably from about 25 to about 40 cm.

A feature that can be useful in defining preferred polymeric foams is the cell structure. Foam cells, and especially cells that are formed by polymerizing a monomer-containing oil phase that surrounds relatively monomer-free water-phase droplets, will frequently be substantially spherical in shape. These spherical cells are connected to each other by openings, which are referred to hereafter as holes between cells. Both the size or "diameter" of such spherical cells and the diameter of the openings (holes) between the cells are commonly used for characterizing foams in general. Since the cells, and holes between the cells, in a given sample of polymeric foam will not necessarily be of approximately the same size; average cell and hole sizes, i.e., average cell and hole diameters, will often be specified.

Cell and hole sizes are parameters that can impact a number of important mechanical and performance features of the, including the liquid wicking properties of these foams, as well as the capillary pressure that is developed within the foam structure. A number of techniques are available for determining the average cell and hole sizes of foams. A useful technique involves a simple

measurement based on the scanning electron photomicrograph of a foam sample. The foams useful as absorbents for aqueous liquids in accordance with the present invention will preferably have a number average cell size of from about 20  $\mu\text{m}$  to about 60  $\mu\text{m}$ , and typically from about 30  $\mu\text{m}$  to about 50  $\mu\text{m}$ , and a number average hole size of from about 5  $\mu\text{m}$  to about 15  $\mu\text{m}$ , and typically from about 8  $\mu\text{m}$  to about 12  $\mu\text{m}$ .

"Capillary suction specific surface area" is a measure of the test-liquid-accessible surface area of the polymeric network accessible to the test liquid. Capillary suction specific surface area is determined both by the dimensions of the cellular units in the foam and by the density of the polymer, and is thus a way of quantifying the total amount of solid surface provided by the foam network to the extent that such a surface participates in absorbency.

For purposes of this invention, capillary suction specific surface area is determined by measuring the amount of capillary uptake of a low surface tension liquid (e.g., ethanol) which occurs within a foam sample of a known mass and dimensions. A detailed description of such a procedure for determining foam specific surface area via the capillary suction method is set forth in the Test Methods section of U.S. Patent No. 5,387,207 *supra*. Any reasonable alternative method for determining capillary suction specific surface area can also be utilized.

Distribution foams useful for the present invention will preferably have a capillary suction specific surface area of at least about 0.01  $\text{m}^2/\text{ml}$ , more preferably at least about 0.03  $\text{m}^2/\text{ml}$ . Typically, the capillary suction specific surface area is in the range from about 0.01 to about 0.20  $\text{m}^2/\text{ml}$ , preferably from about 0.03 to about 0.10  $\text{m}^2/\text{ml}$ , most preferably from about 0.04 to about 0.08  $\text{m}^2/\text{ml}$ .

"Foam density" (i.e., in grams of foam per cubic centimeter of foam volume in air) is specified herein on a dry basis. The density of the foam, like capillary suction specific surface area, can influence a number of performance and mechanical characteristics of absorbent foams. These include the absorbent capacity for aqueous liquids and the compression deflection characteristics.

Foam density will vary according to the state of the foam. Foams in the collapsed state obviously have higher density than the same foam in the fully expanded state. In general, foams in the collapsed state useful for the present invention have a dry density of about  $0.11 \text{ g/cm}^3$ .

Any suitable gravimetric procedure that will provide a determination of mass of solid foam material per unit volume of foam structure can be used to measure foam density. For example, an ASTM gravimetric procedure described more fully in the Test Methods section of U.S. Patent No. 5,387,207 *supra* is one method that can be employed for density determination. Foam density pertains to the weight per unit volume of a washed foam free of emulsifiers, fillers, surface treatments such as salts, and the like. The foams useful for the present invention will preferably have dry densities of from about  $8 \text{ mg/cm}^3$  to about  $77 \text{ mg/cm}^3$ , more preferably from about  $11 \text{ mg/cm}^3$  to about  $63 \text{ mg/cm}^3$ , still more preferably from about  $13 \text{ mg/cm}^3$  to about  $48 \text{ mg/cm}^3$ .

Foams useful for the present invention can be obtained by polymerizing a specific type of water-in-oil emulsion or HIPE having a relatively small amount of an oil phase and a relatively greater amount of a water phase. This process comprises the steps of:

- A) forming a water-in-oil emulsion at a specified temperature and under specified shear mixing from:
  - 1) an oil phase comprising:
    - a) from about 85 to about 98% by weight of a monomer component capable of forming a copolymer having a  $T_g$  of about  $35^\circ\text{C}$  or lower, the monomer component comprising:
      - i) from about 30 to about 80% by weight of at least one substantially water-insoluble monofunctional monomer capable of forming an atactic amorphous polymer having a  $T_g$  of about  $25^\circ\text{C}$  or lower;
      - ii) from about 5 to about 40% by weight of at least one substantially water-insoluble monofunctional comonomer capable of imparting toughness about equivalent to that provided by styrene;
      - iii) from about 5 to about 30% by weight of a first substantially water-insoluble, polyfunctional crosslinking

- agent selected from divinyl benzenes, trivinylbenzenes, divinyltoluenes, divinylxylenes, divinyl-naphthalenes, divinylalkylbenzenes, divinylphenanthrenes, divinylbiphenyls, divinyl-diphenyl-methanes, divinylbenzyls, divinylphenylethers, divinyl-diphenyl-sulfides, divinylfurans, divinylsulfide, divinyl sulfone, and mixtures thereof; and
- iv) from 0 to about 15% by weight of a second substantially water-insoluble, polyfunctional crosslinking agent selected from polyfunctional acrylates, methacrylates, acrylamides, methacryl-amides, and mixtures thereof; and
- b) from about 2 to about 15% by weight of an emulsifier component which is soluble in the oil phase and which is suitable for forming a stable water-in-oil emulsion, the emulsion component comprising: (i) a primary emulsifier having at least about 40% by weight emulsifying components selected from diglycerol monoesters of linear unsaturated C<sub>16</sub>-C<sub>22</sub> fatty acids, diglycerol monoesters of branched C<sub>16</sub>-C<sub>24</sub> fatty acids, diglycerol monoaliphatic ethers of branched C<sub>16</sub>-C<sub>24</sub> alcohols, diglycerol monoaliphatic ethers of linear unsaturated C<sub>16</sub>-C<sub>22</sub> fatty alcohols, diglycerol monoaliphatic ethers of linear saturated C<sub>12</sub>-C<sub>14</sub> alcohols, sorbitan monoesters of linear unsaturated C<sub>16</sub>-C<sub>22</sub> fatty acids, sorbitan monoesters of branched C<sub>16</sub>-C<sub>24</sub> fatty acids, and mixtures thereof; or (ii) the combination a primary emulsifier having at least 20% by weight of these emulsifying components and certain secondary emulsifiers in a weight ratio of primary to secondary emulsifier of from about 50:1 to about 1:4; and
- 2) a water phase comprising an aqueous solution containing: (i) from about 0.2 to about 20% by weight of a water-soluble electrolyte; and (ii) an effective amount of a polymerization initiator;
- 3) a volume to weight ratio of water phase to oil phase in the range of from about 12:1 to about 125:1; and

- B) polymerizing the monomer component in the oil phase of the water-in-oil emulsion to form a polymeric foam material; and
- C) optionally dewatering the polymeric foam material.

The process allows the formation of these absorbent foams that are capable of distributing liquids as a result of having carefully balanced properties as described herein. These properties are achieved by careful selection of crosslinker and monomer types and levels and emulsion formation parameters, specifically the amount of shear mixing, the temperature, and the water-to-oil ratio (which translates into the final density of the dry foam).

Polymeric foams useful for the present invention can be prepared by polymerization of certain water-in-oil emulsions having a relatively high ratio of water phase to oil phase commonly known in the art as "HIPEs". Polymeric foam materials which result from the polymerization of such emulsions are referred to hereafter as "HIPE foams". A detailed description of the general preparation of such HIPEs is given in U.S. Patent No. 5,563,179 and U.S. Patent No. 5,387,207, *infra*.

The relative amounts of the water and oil phases used to form the HIPEs are, among many other parameters, important in determining the structural, mechanical and performance properties of the resulting polymeric foams. In particular, the ratio of water to oil ("W:O ratio") in the emulsion varies inversely with ultimate foam density and can influence the cell size and capillary suction specific surface area of the foam and dimensions of the struts that form the foam. The emulsions used to prepare the HIPE foams useful for this invention will generally have a volume to weight ratio of water phase to oil phase in the range of from about 12:1 to about 125:1, and most typically from about 15:1 to about 90:1. Particularly preferred foams can be made from HIPEs having ratios of from about 20:1 to about 75:1.

The major portion of the oil phase of the HIPEs will comprise monomers, comonomers and crosslinking agents such as those enumerated in U.S. Patent No. 5,387,207 *infra*. It is essential that these monomers, comonomers and crosslinking agents be substantially water-insoluble so that they are primarily soluble in the oil phase and not the water phase. Use of such substantially

water-insoluble monomers ensures that HIPEs of appropriate characteristics and stability will be realized. It is, of course, highly preferred that the monomers, comonomers and crosslinking agents used herein be of the type such that the resulting polymeric foam is suitably non-toxic and appropriately chemically stable. These monomers, comonomers and cross-linking agents should preferably have little or no toxicity if present at very low residual concentrations during post-polymerization foam processing and/or use.

Another essential component of the oil phase is an emulsifier component that permits the formation of stable HIPEs. This emulsifier component comprises a primary emulsifier and optionally a secondary emulsifier, such as those enumerated in U.S. Patent No. 5,387,207 *infra*.

The oil phase used to form the HIPEs comprises from about 85 to about 98% by weight monomer component and from about 2 to about 15% by weight emulsifier component. Preferably, the oil phase will comprise from about 90 to about 98% by weight monomer component and from about 3 to about 10% by weight emulsifier component. The oil phase also can contain other optional components. One such optional component is an oil soluble polymerization initiator of the general type well known to those skilled in the art, such as described in U.S. Patent No. 5,290,820 (Bass et al.), issued March 1, 1994, which is incorporated by reference. Another preferred optional component is an antioxidant such as a Hindered Amine Light Stabilizer (HALS) and Hindered Phenolic Stabilizers (HPS) or any other antioxidant compatible with the initiator system to be employed. Other optional components include plasticizers, fillers, colorants, chain transfer agents, dissolved polymers, and the like.

The discontinuous water internal phase of the HIPE is generally an aqueous solution containing one or more dissolved components such as those enumerated in U.S. Patent No. 5,387,207 *infra*. One essential dissolved component of the water phase is a water-soluble electrolyte. The dissolved electrolyte minimizes the tendency of the monomers, comonomers and crosslinkers that are primarily oil soluble to also dissolve in the water phase.

This, in turn, is believed to minimize the extent to which polymeric material fills the cell windows at the oil/water interfaces formed by the water phase droplets during polymerization. Thus, the presence of electrolyte and the

resulting ionic strength of the water phase is believed to determine whether and to what degree the resulting preferred polymeric foams can be open-celled.

The HIPEs will also typically contain a polymerization initiator. Such an initiator component is generally added to the water phase of the HIPEs and can be any conventional water-soluble free radical initiator. These include peroxygen compounds such as sodium, potassium and ammonium persulfates, hydrogen peroxide, sodium peracetate, sodium percarbonate and the like. Conventional redox initiator systems can also be used. Such systems are formed by combining the foregoing peroxygen compounds with reducing agents such as sodium bisulfite, L-ascorbic acid or ferrous salts.

The initiator can be present at up to about 20 mole percent based on the total moles of polymerizable monomers present in the oil phase. More preferably, the initiator is present in an amount of from about 0.001 to about 10 mole percent based on the total moles of polymerizable monomers in the oil phase.

The polymer forming the HIPE foam structure will preferably be substantially free of polar functional groups. This means the polymeric foam will be relatively hydrophobic in character. These hydrophobic foams can find utility where the absorption of hydrophobic liquids is desired. Uses of this sort include those where an oily component is mixed with water and it is desired to separate and isolate the oily component, such as in the case of marine oil spills.

When these foams are to be used as absorbents for aqueous liquids such as juice spills, milk, and the like for clean up and/or bodily liquids such as urine, they generally require further treatment to render the foam relatively more hydrophilic. Hydrophilization of the foam, if necessary, can generally be accomplished by treating the HIPE foam with a hydrophilizing surfactant in a manner described in U.S. Patent No. 5,387,207 *infra*.

These hydrophilizing surfactants can be any material that enhances the water wettability of the polymeric foam surface. They are well known in the art, and can include a variety of surfactants, preferably of the nonionic type, such as those enumerated in U.S. Patent No. 5,387,207 *infra*.

Another material that is typically incorporated into the HIPE foam structure is a hydratable, and preferably hygroscopic or deliquescent, water soluble inorganic salt. Such salts include, for example, toxicologically acceptable alkaline earth metal salts. Salts of this type and their use with oil-soluble surfactants as the foam hydrophilizing surfactant is described in greater detail in U.S. Patent No. 5,352,711 (DesMarais), issued October 4, 1994, the disclosure of which is incorporated by reference. Preferred salts of this type include the calcium halides such as calcium chloride that, as previously noted, can also be employed as the water phase electrolyte in the HIPE.

Hydratable inorganic salts can easily be incorporated by treating the foams with aqueous solutions of such salts. These salt solutions can generally be used to treat the foams after completion of, or as part of, the process of removing the residual water phase from the just-polymerized foams. Treatment of foams with such solutions preferably deposits hydratable inorganic salts such as calcium chloride in residual amounts of at least about 0.1% by weight of the foam, and typically in the range of from about 0.1 to about 12%.

Treatment of these relatively hydrophobic foams with hydrophilizing surfactants (with or without hydratable salts) will typically be carried out to the extent necessary to impart suitable hydrophilicity to the foam. Some foams of the preferred HIPE type, however, are suitably hydrophilic as prepared, and can have incorporated therein sufficient amounts of hydratable salts, thus requiring no additional treatment with hydrophilizing surfactants or hydratable salts. In particular, such preferred HIPE foams include those where certain oil phase emulsifiers previously described and calcium chloride are used in the HIPE. In those instances, the internal polymerized foam surfaces will be suitably hydrophilic, and will include residual water-phase liquid containing or depositing sufficient amounts of calcium chloride, even after the polymeric foams have been dewatered to a practicable extent.

Foam preparation typically involves the steps of: 1) forming a stable high internal phase emulsion (HIPE); 2) polymerizing/curing this stable emulsion under conditions suitable for forming a solid polymeric foam structure; 3) optionally washing the solid polymeric foam structure to remove the original residual water phase from the polymeric foam structure and, if necessary,



treating the polymeric foam structure with a hydrophilizing surfactant and/or hydratable salt to deposit any needed hydrophilizing surfactant/hydratable salt, and 4) thereafter dewatering this polymeric foam structure. The procedure is described more fully in U.S. Patent No. 5,387,207 *supra*.

#### Absorbent Articles

The materials according to the present invention are suitable for an number of applications requiring fluid transport (i.e. receiving, moving, releasing), but are particularly useful for absorbent articles.

In order to use respective materials in absorbent structures, these materials can be combined with other elements so as to creating an Fluid handling member, which comprises materials according to the description as laid out in the above.

An absorbent article generally comprises:

- an absorbent core or core structure (which comprises the improved fluid distribution members according to the present invention, and which may consist of sub-structures);
- a fluid pervious topsheet;
- a fluid impervious backsheet;
- optionally further features like closure elements or elastification.

Figure 6 is a plan view of an exemplary embodiment of an absorbent article of the invention which is a diaper.

The diaper 20 is shown in Figure 6 in its flat-out, uncontracted state (i.e. with elastic induced contraction pulled out except in the side panels wherein the elastic is left in its relaxed condition) with portions of the structure being cut-away to more clearly show the construction of the diaper 20 and with the portion of the diaper 20 which faces away from the wearer, the outer surface 52, facing the viewer. As shown in Figure 6, the diaper 20 comprises a containment assembly 22 preferably comprising a liquid pervious topsheet 24, a liquid impervious backsheet 26 joined with the topsheet 24, and an absorbent core 28 positioned between the topsheet 24 and the backsheet 26; elasticized side panels 30; elasticized leg cuffs 32; an elastic waist feature 34; and a closure system comprising a dual tension fastening system generally multiply designated as 36.

The dual tension fastening system 36 preferably comprises a primary fastening system 38 and a waist closure system 40. The primary fastening system 38 preferably comprises a pair of securement members 42 and a landing member 44. The waist closure system 40 is shown in Figure 6 to preferably comprise a pair of first attachment components 46 and a second attachment component 48. The diaper 20 also preferably comprises a positioning patch 50 located subjacent each first attachment component 46.

The diaper 20 is shown in Figure 6 to have an outer surface 52 (facing the viewer in Figure 6), an inner surface 54 opposed to the outer surface 52, a first waist region 56, a second waist region 58 opposed to the first waist region 56, and a periphery 60 which is defined by the outer edges of the diaper 20 in which the longitudinal edges are designated 62 and the end edges are designated 64. The inner surface 54 of the diaper 20 comprises that portion of the diaper 20 which is positioned adjacent to the wearer's body during use (i.e. the inner surface 54 generally is formed by at least a portion of the topsheet 24 and other components joined to the topsheet 24). The outer surface 52 comprises that portion of the diaper 20 which is positioned away from the wearer's body (i.e. the outer surface 52 generally is formed by at least a portion of the backsheet 26 and other components joined to the backsheet 26). The first waist region 56 and the second waist region 58 extend, respectively, from the end edges 64 of the periphery 60 to the lateral centreline 66 of the diaper 20. The waist regions each comprise a central region 68 and a pair of side panels which typically comprise the outer lateral portions of the waist regions. The side panels positioned in the first waist region 56 are designated 70 while the side panels in the second waist region 58 are designated 72. While it is not necessary that the pairs of side panels or each side panel be identical, they are preferably mirror images one of the other. The side panels 72 positioned in the second waist region 58 can be elastically extensible in the lateral direction (i.e. elasticized side panels 30). (The lateral direction (x direction or width) is defined as the direction parallel to the lateral centreline 66 of the diaper 20; the longitudinal direction (y direction or length) being defined as the direction parallel to the longitudinal centreline 67; and the axial direction (Z direction or thickness) being defined as the direction extending through the thickness of the diaper 20).

Figure 6 shows a specific of the diaper 20 in which the topsheet 24 and the backsheet 26 have length and width dimensions generally larger than those of

the absorbent core 28. The topsheet 24 and the backsheet 26 extend beyond the edges of the absorbent core 28 to thereby form the periphery 60 of the diaper 20. The periphery 60 defines the outer perimeter or, in other words, the edges of the diaper 20. The periphery 60 comprises the longitudinal edges 62 and the end edges 64.

While each elasticized leg cuff 32 may be configured so as to be similar to any of the leg bands, side flaps, barrier cuffs, or elastic cuffs described above, it is preferred that each elasticized leg cuff 32 comprise at least an inner barrier cuff 84 comprising a barrier flap 85 and a spacing elastic member 86 such as described in the above-referenced US Patent 4,909,803. In a preferred embodiment, the elasticized leg cuff 32 additionally comprises an elastic gasketing cuff 104 with one or more elastic strands 105, positioned outboard of the barrier cuff 84 such as described in the above-references US Patent 4,695,278.

The diaper 20 may further comprise an elastic waist feature 34 that provides improved fit and containment. The elastic waist feature 34 at least extends longitudinally outwardly from at least one of the waist edges 83 of the absorbent core 28 in at least the central region 68 and generally forms at least a portion of the end edge 64 of the diaper 20. Thus, the elastic waist feature 34 comprises that portion of the diaper at least extending from the waist edge 83 of the absorbent core 28 to the end edge 64 of the diaper 20 and is intended to be placed adjacent the wearer's waist. Disposable diapers are generally constructed so as to have two elastic waist features, one positioned in the first waist region and one positioned in the second waist region.

The elasticized waist band 35 of the elastic waist feature 34 may comprise a portion of the topsheet 24, a portion of the backsheet 26 that has preferably been mechanically stretched and a bi-laminate material comprising an elastomeric member 76 positioned between the topsheet 24 and backsheet 26 and resilient member 77 positioned between backsheet 26 and elastomeric member 76.

This as well as other components of the diaper are given in more detail in WO 93/16669 which is incorporated herein by reference.

### Absorbent core

The absorbent core should be generally compressible, conformable, non-irritating to the wearer's skin, and capable of absorbing and retaining liquids such as urine and other certain body exudates. As shown in Figure 6, the absorbent core has a garment surface ("lower" or "bottom" part), a body surface, side edges, and waist edges. The absorbent core may - in addition to the fluid distribution member according to the present invention - comprise a wide variety of liquid-absorbent or liquid handling materials commonly used in disposable diapers and other absorbent articles such as - but not limited to - comminuted wood pulp which is generally referred to as airfelt; meltblown polymers including coform; chemically stiffened, modified or cross-linked cellulosic fibers; tissue including tissue wraps and tissue laminates.

General examples for absorbent structures are described in U.S. Patent 4,610,678 entitled "High-Density Absorbent Structures" issued to Weisman et al. on September 9, 1986; U.S. Patent 4,673,402 entitled "Absorbent Articles With Dual-Layered Cores" issued to Weisman et al. on June 16, 1987; U.S. Patent 4,888,231 entitled "Absorbent Core Having A Dusting Layer" issued to Angstadt on December 19, 1989; EP-A-0 640 330 of Bewick-Sonntag et al.; US 5 180 622 (Berg et al.); US 5 102 597 (Roe et al.); US 5 387 207 (LaVon). Such and similar structures might be adopted to be compatible with the requirements outlined below for being used as the absorbent core 28.

The absorbent core can be a unitary core structure, or it can be a combination of several absorbent structures, which in turn can consist of one or more sub-structures. Each of the structures or sub-structures can have an essentially two-dimensional extension (i.e. be a layer) or a three-dimensional shape.

### Regions of absorbent articles

Generally, absorbent hygienic articles are intended for being worn around the lower end of the body torso. It is an essential design feature of these articles to cover the regions of the body where the discharges occur ("discharge regions"), which extend around the respective body openings. The respective zones of the absorbent article covering the discharge regions are correspondingly referred to as "loading zones". Thus during use, the articles are generally arranged on the

wearer such that they extend (for a standing position of the wearer) from the crotch between the legs upwards, both in the front and the back of the wearer.

Generally, such articles have a length dimension exceeding their width dimension, whereby the article is worn such that the axis of the length dimension is aligned with the height direction of the wearer when standing, whilst the width direction of the article is aligned with a line extending from left to right of the wearer.

Because of the anatomy of the human wearer, the space between the legs of the wearer generally confines the space available for the article in this region. For good fit, an absorbent article should be designed such that it fits well in the crotch region. If the width of the article is excessively wide relative to the crotch width of the wearer, the article may be deformed, which might result in deteriorated performance, and reduced wearers comfort.

The point, where the article has its smallest width to fit best between the legs of the wearer then coincides with the point on the wearer, where the distance between the legs is the narrowest, and is - for the scope of the present invention - referred to as the "crotch point".

If the crotch point of an article is not obvious from its shape, it can be determined by placing the article on a wearer of the intended user group (e.g. a toddler) preferably in a standing position, and then placing an extensible filament around the legs in a figure eight configuration. The point in the article corresponding to the point of intersection of the filament is deemed to be the crotch point of the article and consequently also of the absorbent core being affixed within this article.

Whilst this crotch point of the article is often in the middle of the article (in longitudinal direction) this is not necessarily the case. It can very well be, that the part of the article which is intended to be worn in the front is smaller than the back (or rear) part - either in its length dimension, or width, or both, or surface area. Also, the crotch point does not need to be positioned in the middle of the absorbent core, in particular when the absorbent core is not placed longitudinally centred within the article.

The crotch region is the area surrounding the crotch point, so as to cover the respective body openings, respectively discharge regions. Unless otherwise mentioned, this region extends over a length of 50% of the total core length (which, in turn is defined as the distance between the front and rear waist edges of the core, which might be approximated by straight lines perpendicular to the longitudinal center line). If the crotch point is positioned in the middle of the article, then the crotch region starts (when counting from the front core edge) at 25% of total length and extends up to 75% of the total core length. Or, the front and the rear quarter of the length of the absorbent core do not belong to the crotch region, the rest does.

The crotch region length being 50% of the total absorbent core length has been derived for baby diapers, where it has been confirmed that this is a suitable means to describe the fluid handling phenomena. If the present invention is applied in articles having drastically different dimensions, it might become necessary to reduce these 50% (as in the case for Severe Incontinence articles) or to increase this ratio (as in the case for Ultra Light or Light Incontinence articles). In more general terms, this crotch region of the article should not extend much beyond the discharge region of the wearer.

If the crotch point is positioned offset from the mid-point of the article, the crotch region still covers 50% of the total article length (in longitudinal direction), however, not evenly distributed between front and back, but proportionally adjusted to this off-set.

As an example for an article having a total core length of 500 mm, and having a crotch point which is positioned centered, the crotch region will extend from 125 mm away from the front edge up to 375 mm away from front edge. Or, if the crotch point lies 50 mm offset towards the front core edge, (i.e. being 200 mm away from front core edge), the crotch region extends from 100 mm to 350 mm.

In general terms, for an article having a total core length of  $L_c$ , a crotch point being at a distance  $L_{cp}$  away from the front core edge, and a crotch zone length of  $L_{cz}$ , the front edge of said crotch zone will be positioned at a distance

$$L_{fecz} = L_{cp} * (1 - L_{cz} / L_c).$$

For example the absorbent article can be a baby diaper, for being worn by toddlers (i.e. of about 12 to 18 kg baby weight) whereby the size of the article in the trade is generally referred to as MAXI size. Then the article has to be able to receive and retain both fecal materials and urine, whereas for the context of the present invention the crotch region has to be capable to primarily receive urine loadings.

The total area and size of the crotch region is - of course - also depending on the respective width of the absorbent core, i.e. if the core is narrower in the crotch region than outside the crotch region, the crotch region has a smaller area (surface) than the remaining area of the absorbent core.

Whilst it can be contemplated, that the boundaries between crotch region and the rest of the article can also be curvilinear, they are approximated within the present description to be straight lines, perpendicular to the longitudinal axis of the article.

The "crotch region" is further confined by the width of the core in this respective region, and the "crotch region area" by the surface as being defined by the crotch region length and the respective width.

As a complementary element to the crotch region, the absorbent core also comprises at least one but mostly two waist region(s), extending towards the front and/or the rear of the absorbent core outside the crotch region.

#### Design Capacity and Ultimate Storage Capacity

In order to be able to compare absorbent articles for varying end use conditions, or differently sized articles, the "design capacity" has been found to be a suitable measure.

For example, babies are representing a typical usage group, but even within this group the amount of urine loading, frequency of loading, composition of the

urine will vary widely from smaller babies (new-born babies) to toddlers on one side, but also for example among various individual babies.

Another user group may be larger children, still suffering from a certain form of incontinence.

Also, incontinent adults can use such articles, again with a wide range of loading conditions, generally referred to as light incontinence ranging up to severe incontinence.

Whilst the man skilled in the art will readily be able to transfer the teaching to other sizes for further discussion, focus will be put on the toddler sized babies. For such user, urine loadings of up to 75 ml per voiding, with on an average of four voidings per wearing period resulting in a total loading of 300 ml, and voiding rates of 15 ml/sec have been found to be sufficiently representative.

Henceforth, such articles being able to cope with such requirements should have the capability of picking up such amounts of urine, which will be referred to for the further discussion as "design capacity".

These amounts of fluids have to be absorbed by materials which can ultimately store the bodily fluids, or at least the aqueous parts of these, such that - if any - only little fluid is left on the surface of the article towards the wearers skin. The term "ultimate" refers in one respect to the situation as in the absorbent article at long wearing times, in the other respect to absorbent materials which reach their "ultimate" capacity when being equilibrated with their environment. This can be in such an absorbent article under real in-use conditions after long wearing times, or this also can be in a test procedure for pure materials or material composites. As many of the processes under consideration have asymptotic kinetic behavior, one skilled in the art will readily consider "ultimate" capacities to be reached when the actual capacity has reached a value sufficiently close to the asymptotic endpoint, e.g. relative to the equipment measurement accuracy.

As an absorbent article can comprise materials which are primarily designed to ultimately store fluids, and other materials which are primarily designed to fulfill other functions such as acquisition and/or distribution of the fluid, but may still



have a certain ultimate storage capability, suitable core materials according to the present invention are described without attempting to artificially separate such functions. Nonetheless, the ultimate storage capacity can be determined for the total absorbent core, for regions thereof, for absorbent structures, or even sub-structures, but also for materials as being used in any of the previous.

As discussed in the above for varying the dimensions of the article, one skilled in the art will be able to readily adopt the appropriate design capacities for other intended user groups.

#### Materials for being used in absorbent cores

The absorbent core can comprise fibrous materials to form fibrous web or fibrous matrices.

Useful fibers include those that are naturally occurring fibers (modified or unmodified), as well as synthetically made fibers. Examples of suitable unmodified/modified naturally occurring fibers include cotton, Esparto grass, bagasse, kemp, flax, silk, wool, wood pulp, chemically modified wood pulp, jute, rayon, ethyl cellulose, and cellulose acetate. Suitable synthetic fibers can be made from polyvinyl chloride, polyvinyl fluoride, polytetrafluoroethylene, polyvinylidene chloride, polyacrylics such as ORLON®, polyvinyl acetate, polyethylvinyl acetate, non-soluble or soluble polyvinyl alcohol, polyolefins such as polyethylene (e.g., PULPEX®) and polypropylene, polyamides such as nylon, polyesters such as DACRON® or KODEL®, polyurethanes, polystyrenes, and the like. The fibers used can comprise solely naturally occurring fibers, solely synthetic fibers, or any compatible combination of naturally occurring and synthetic fibers. The fibers used in the present invention can be hydrophilic, or can be a combination of both hydrophilic and hydrophobic fibers.

For many absorbent cores or core, the use of hydrophilic fibers is preferred. Suitable hydrophilic fibers include cellulosic fibers, modified cellulosic fibers, rayon, polyester fibers such as polyethylene terephthalate (e.g., DACRON®), hydrophilic nylon (HYDROFIL®), and the like. Suitable hydrophilic fibers can also be obtained by hydrophilizing hydrophobic fibers, such as surfactant-treated or silica-treated thermoplastic fibers derived from, for example, polyolefins such as polyethylene or polypropylene, polyacrylics, polyamides, polystyrenes, polyurethanes and the like.

Suitable wood pulp fibers can be obtained from many well-known chemical processes such as - but not limited to - the Kraft and sulfite processes. A further suitable type of fibers is chemically stiffened cellulose. As used herein, the term "chemically stiffened cellulosic fibers" means cellulosic fibers that have been stiffened by chemical means to increase the stiffness of the fibers under both dry and aqueous conditions. Such means can include the addition of a chemical stiffening agent that, for example, coats and/or impregnates the fibers. Such means can also include the stiffening of the fibers by altering the chemical structure, e.g., by crosslinking polymer chains.

Polymeric stiffening agents that can coat or impregnate the cellulosic fibers include: cationic modified starches having nitrogen-containing groups (e.g., amino groups) such as those available from National Starch and Chemical Corp., Bridgewater, NJ, USA; latexes; wet strength resins such as polyamide-epichlorohydrin resin (e.g., Kymene® 557H, Hercules, Inc. Wilmington, Delaware, USA), polyacrylamide resins described, for example, in U.S. Patent 3,556,932 (Coscia et al), issued January 19, 1971; commercially available polyacrylamides marketed by American Cyanamid Co., Stamford, CT, USA, under the tradename Parex® 631 NC; urea formaldehyde and melamine formaldehyde resins, and polyethylenimine resins.

These fibers can also be stiffened by chemical reaction. For example, crosslinking agents can be applied to the fibers that, subsequent to application, are caused to chemically form intrafiber crosslink bonds. These crosslink bonds can increase the stiffness of the fibers. While the utilisation of intrafiber crosslink bonds to chemically stiffen the fiber is preferred, it is not meant to exclude other types of reactions for chemical stiffening of the fibers.

Fibers stiffened by crosslink bonds in individualised form (i.e., the individualised stiffened fibers, as well as process for their preparation) are disclosed, for example, in U.S. Patent 3,224,926 (Bernardin), issued December 21, 1965; U.S. Patent 3,440,135 (Chung), issued April 22, 1969; U.S. Patent 3,932,209 (Chatterjee), issued January 13, 1976; and U.S. Patent 4,035,147 (Sangenis et al), issued December 19, 1989; U.S. Patent 4,898,642d (Moore et al), issued February 6, 1990; and U.S. Patent 5,137,537 (Herron et al), issued August 11, 1992.

In currently preferred stiffened fibers, chemical processing includes intrafiber crosslinking with crosslinking agents while such fibers are in a relatively dehydrated, defibrated (i.e., individualised), twisted, curled condition. Suitable chemical stiffening agents are typically monomeric crosslinking agents including, especially C<sub>2</sub>-C<sub>9</sub> polycarboxylic acids such as citric acid. Preferably, such stiffened fibers are twisted and curled as described in more details in U.S. Patent 4,898,642.

These chemically stiffened cellulosic fibers have certain properties that make them particularly useful in certain absorbent, relative to unstiffened cellulosic fibers. In addition to being hydrophilic, these stiffened fibers have unique combinations of stiffness and resiliency.

In addition to or alternatively synthetic or thermoplastic fibers can comprised in the absorbent structures, such as being made from any thermoplastic polymer that can be melted at temperatures that will not extensively damage the fibers. Preferably, the melting point of this thermoplastic material will be less than about 190°C, and preferably between about 75°C and about 175°C. In any event, the melting point of this thermoplastic material should be no lower than the temperature at which the thermally bonded absorbent structures, when used in absorbent articles, are likely to be stored. The melting point of the thermoplastic material is typically no lower than about 50°C.

The thermoplastic materials, and in particular the thermoplastic fibers, can be made from a variety of thermoplastic polymers, including polyolefins such as polyethylene (e.g., PULPEX®) and polypropylene, polyesters, copolyesters, polyvinyl acetate, polyamides, copolyamides, polystyrenes, polyurethanes and copolymers of any of the foregoing such as vinyl chloride/vinyl acetate, and the like. Suitable thermoplastic materials include hydrophobic fibers that have been made hydrophilic, such as surfactant-treated or silica-treated thermoplastic fibers derived from, for example, polyolefins such as polyethylene or polypropylene, polyacrylics, polyamides, polystyrenes, polyurethanes and the like. The surface of the hydrophobic thermoplastic fiber can be rendered hydrophilic by treatment with a surfactant, such as a nonionic or anionic surfactant, e.g., by spraying the fiber with a surfactant, by dipping the fiber into a surfactant or by including the

surfactant as part of the polymer melt in producing the thermoplastic fiber. Upon melting and resolidification, the surfactant will tend to remain at the surfaces of the thermoplastic fiber. Suitable surfactants include nonionic surfactants such as Brij® 76 manufactured by ICI Americas, Inc. of Wilmington, Delaware, and various surfactants sold under Pegosperse® trademark by Glyco Chemical, Inc. of Greenwich, Connecticut. Besides nonionic surfactants, anionic surfactants can also be used. These surfactants can be applied to the thermoplastic fibers at levels of, for example, from about 0.2 to about 1 gram per square of centimeter of thermoplastic fiber.

Suitable thermoplastic fibers can be made from a single polymer (monocomponent fibers), or can be made from more than one polymer (e.g., bicomponent fibers). For example, "bicomponent fibers" can refer to thermoplastic fibers that comprise a core fiber made from one polymer that is encased within a thermoplastic sheath made from a different polymer. The polymer comprising the sheath often melts at a different, typically lower, temperature than the polymer comprising the core. As a result, these bicomponent fibers provide thermal bonding due to melting of the sheath polymer, while retaining the desirable strength characteristics of the core polymer.

Suitable bicomponent fibers can include sheath/core fibers having the following polymer combinations: polyethylene/polypropylene, polyethylvinyl acetate/polypropylene, poly-ethylene/polyester, polypropylene/polyester, copolyester/polyester, and the like. Particularly suitable bicomponent thermoplastic fibers for use herein are those having a polypropylene or polyester core, and a lower melting copolyester, polyethylvinyl acetate or polyethylene sheath (e.g., DANAKLON®, CELBOND® or CHISSO® bicomponent fibers). These bicomponent fibers can be concentric or eccentric. As used herein, the terms "concentric" and "eccentric" refer to whether the sheath has a thickness that is even, or uneven, through the cross-sectional area of the bicomponent fiber. Eccentric bicomponent fibers can be desirable in providing more compressive strength at lower fiber thicknesses. Suitable bicomponent fibers for use herein can be either uncrimped (i.e. bent). Bicomponent fibers can be crimped by typical textile means such as, for example, a stuffer boy method or the gear crimp method to achieve a predominantly two-dimensional or "flat" crimp.

In the case of thermoplastic fibers, their length can vary depending upon the particular melt point and other properties desired for these fibers. Typically, these thermoplastic fibers have a length from about 0.3 to about 7.5 cm long, preferably from about 0.4 to about 3.0 cm long. The properties, including melt point, of these thermoplastic fibers can also be adjusted by varying the diameter (caliper) of the fibers. The diameter of these thermoplastic fibers is typically defined in terms of either denier (grams per 9000 meters) or decitex (grams per 10,000 meters dtex). Depending on the specific arrangement within the structure, suitable thermoplastic fibers can have a decitex in the range from well below 1 decitex, such as 0.4 decitex to about 20 dtex.

Said fibrous materials may be used in an individualised form when the absorbent article is being produced, and an airlaid fibrous structure is formed on the line. Said fibers may also be used as a performed fibrous web or tissue. These structures are then delivered to the production of the article essentially in endless or very long form (e.g. on a roll, spool) and will then be cut to the appropriate size. This can be done on each of such materials individually before these are combined with other materials to form the absorbent core, or when the core itself is cut and said materials are co-extensive with the core.

There is a wide variety of making such web, and such processes are very well known in the art.

With regard to fibers used for producing such webs, there is nearly no limitation in principle - though certain specific web forming and bonding processes might not be fully compatible with certain materials or fiber types.

When looking at individualised fibers as a starting material for making a web, these can be laid down in a fluid medium - if this is gaseous (air) such structures are generally referred to as "dry-laid", if it is liquid such structures are generally referred to as "wet-laid". "Wet-laying" is broadly used to produce paper tissues with a wide range of properties. This term is most commonly used with cellulosic materials, however, also synthetic fibers can be included.

"Dry-laying" is broadly used for non-woven webs, and often the carding process can be used to form such webs. Also the commonly known "air-laid tissues" fall under this category.

A molten polymer can be extruded into fibers which then can be formed directly into a web (i.e. omitting the process step of making individual fibers which then are formed into a web in a separate process step). The resulting structures are commonly referred to as non-wovens of the meltblown type or - if fibers are significantly more drawn - spunbonded webs.

Further, webs can also be formed by combining one or more of the other formation technologies.

In order to give certain strength and integrity properties to the web structures, these are generally bonded. The most broadly used technologies are (a) chemical bonding or (b) thermo bonding by melting a part of the web such. For the latter, the fibers can be compressed, resulting in distinct bonding points, which, for example for nonwoven materials, can cover a significant portion of the total area, values of 20% are not uncommon. Or - particularly useful for structures where low densities are desired - "air-through" bonding can be applied, where parts of the polymers e.g. the sheath material of a BiCo-fibers are molten by means of heated air passing through the (often air-laid) web.

After the webs are formed and bonded, these can be further treated to modify specific properties. This can be - as one of many possible examples - additional surfactant to render hydrophobic fibers more hydrophilic, or vice versa. Also, post formation mechanical treatment, such as disclosed in EP application 96108427.4 can be used to impart particularly useful properties to such materials.

Preferably the absorbent cores may comprise in addition or alternatively to fibrous webs, other porous materials, such as foams, in particular open-celled absorbent polymeric foam materials as being described in the above.

#### Superabsorbent polymers or hydrogels

Optionally, and often preferably, the absorbent structures according to the present invention can comprise superabsorbent polymers, or hydrogels. The

hydrogel-forming absorbent polymers useful in the present invention include a variety of substantially water-insoluble, but water-swellaable polymers capable of absorbing large quantities of liquids. Such polymer materials are also commonly referred to as "hydrocolloids", or "superabsorbent" materials. These hydrogel-forming absorbent polymers preferably have a multiplicity of anionic, functional groups, such as sulfonic acid, and more typically carboxy, groups. Examples of polymers suitable for use herein include those which are prepared from polymerisable, unsaturated, acid-containing monomers.

Some non-acid monomers can also be included, usually in minor amounts, in preparing the hydrogel-forming absorbent polymers herein. Such non-acid monomers can include, for example, the water-soluble or water-dispersible esters of the acid-containing monomers, as well as monomers that contain no carboxylic or sulfonic acid groups at all. Examples for such well known materials are described e.g. in U.S. Patent 4,076,663 (Masuda et al), issued February 28, 1978, and in U.S. Patent 4,062,817 (Westerman), issued December 13, 1977.

Hydrogel-forming absorbent polymers suitable for the present invention contain carboxy groups. These polymers include hydrolysed starch-acrylonitrile graft copolymers, partially neutralised starch-acrylonitrile graft copolymers, starch-acrylic acid graft copolymers, partially neutralised starch-acrylic acid graft copolymers, saponified vinyl acetate-acrylic ester copolymers, hydrolysed acrylonitrile or acrylamide copolymers, slightly network crosslinked polymers of any of the foregoing copolymers, partially neutralised polyacrylic acid, and slightly network crosslinked polymers of partially neutralised polyacrylic acid. These polymers can be used either solely or in the form of a mixture of two or more different polymers. Examples of these polymer materials are disclosed in U.S. Patent 3,661,875, U.S. Patent 4,076,663, U.S. Patent 4,093,776, U.S. Patent 4,666,983, and U.S. Patent 4,734,478.

Most preferred polymer materials for use in making hydrogel-forming particles are slightly network crosslinked polymers of partially neutralised polyacrylic acids and starch derivatives thereof. Most preferably, the hydrogel-forming particles comprise from about 50 to about 95%, preferably about 75%, neutralised, slightly network crosslinked, polyacrylic acid (i.e. poly (sodium acrylate/acrylic acid)).

As described above, the hydrogel-forming absorbent polymers are preferably slightly network crosslinked. Network crosslinking serves to render the polymer substantially water-insoluble and, in part, determines the absorptive capacity and extractable polymer content characteristics of the precursor particles and the resultant macrostructures. Processes for network crosslinking the polymers and typical network crosslinking agents are described in greater detail in the herein before-referenced U.S. Patent 4,076,663, and in DE-A-4020780 (Dahmen).

The superabsorbent materials can be used in particulate form or in fibrous form and may also be combined other elements to form preformed structures.

Whilst the individual elements have been disclosed separately, an absorbent structure or substructure can be made by combining one or more of these elements.

Without intending a limiting effect, the following describes suitable combinations.

- i) - Particular Superabsorbent polymer (SAP) mixed with cellulosic or other fibers. The basic principle is well established and known, however, upon attempting to reduce thinness of the articles, higher and higher ratios of weight of SAP to fibers have been employed recently. Within this scope, combination of the SAP with binders such as hot-melt adhesives (such as disclosed in EP-A-0.695.541) or with meltable polymeric material (such as PE particles) can be a suitable tool to immobilise the SAP;
- ii) SAP forming a substructure by interparticle crosslinks, such as described in US
- iii) Fibrous SAP being mixed with other fibers, or forming a fibrous SAP web, such as described in US/EP
- iv) Foam structures comprising superabsorbent particulate materials, such as described in US/EP

#### Examples

These examples illustrate the specific preparation of collapsed HIPE foams useful for the present invention.



Samples 1, 2, 3 - HIPEs as Distribution material

The following Samples A.5 to A.7 are of the polymeric foam type, and are prepared as described generally in the Examples section of U.S. Patent No. 5,563,179, supra. Generally, this process comprises appropriate mixing of an aqueous phase containing selected salts with an oil phase containing selected monomers and emulsifiers. The aqueous phase typically contains an initiator such as potassium persulfate and inorganic salt such as calcium chloride. The oil phase typically contains a blend of monomers such as 2-ethylhexylacrylate and crosslinking monomers such as divinyl benzene (which contains ethyl styrene as an impurity) and 1,6-hexanedioldiacrylate. Adjuvants such as antioxidants, opacifying agents, pigments, dyes, fillers, and other generally unreactive chemicals, can also be added to either phase.

The separate streams of the oil phase and water phase (typically heated to between about 30° and about 90°C) are fed to a dynamic mixing apparatus. Thorough mixing of the combined streams in the dynamic mixing apparatus is achieved by means of a pin impeller. The ratio of the aqueous phase and the oil phase, referred to as the "water-to-oil ratio", or W:O, is used to control the density of the ultimate foam produced. A detailed description of the apparatus and the procedures for establishing the initial HIPE formation is described in more detail in the Examples section of U.S. Patent No. 5,563,179, supra.

Once the apparatus set-up is filled, agitation is begun in the dynamic mixer, with the impeller turning at a specified RPM. The flow rate of the water phase is then steadily increased to a rate of 44.1 cm<sup>3</sup>/sec in a time period of about 30 sec. and the oil phase flow rate is reduced to 1.25 g/sec over a time period of about 1 min. The back pressure created by the dynamic and static mixers at this point is typically between about 3 and about 8 PSI (about 21 to about 55 kPa). The impeller speed is then adjusted to the desired RPM over a period of 120 sec. The system back pressure responds to this adjustment and remains constant thereafter.

The HIPE from the static mixer is collected in a round polypropylene tub, 17 in. (43 cm) in. diameter and 7.5 in. (10 cm) high, with a concentric insert made of Celcon plastic. The insert is 5.0 in. (12.7 cm) in diameter at its base and 4.75 in. (12 cm) in diameter at its top and is 6.75 in. (17.1 cm) high. The HIPE-containing

tubs are kept in a room maintained at 65°C for 18 hours to cure and provide a polymeric HIPE foam.

The cured HIPE foam is removed from the tubs. The foam at this point contains residual water phase (containing dissolved emulsifiers, electrolyte, initiator residues, and initiator). The foam is sliced with a sharp reciprocating saw blade into sheets of desired thickness. These sheets are then subjected to compression in a series of 2 porous nip rolls equipped with vacuum which gradually reduces the residual water phase content of the foam to about 2 times (2X) the weight of the polymerized monomers. At this point, the sheets are then resaturated with a 4% CaCl<sub>2</sub> solution at 60°C, are squeezed in a series of 3 porous nip rolls equipped with vacuum to a water phase content of about 2X. The CaCl<sub>2</sub> content of the foam is between 2 and 10%.

The HIPE foam is then dried in air for about 16 hours or thermally dried continuously. Such drying reduces the moisture content to about 4-20% by weight of polymerized material.

#### Sample 1

Anhydrous calcium chloride (36.32 kg) and potassium persulfate (189 g) are dissolved in 378 liters of water. This provides the water phase stream to be used in a continuous process for forming a HIPE emulsion.

To a monomer combination comprising distilled divinylbenzene (39% divinylbenzene and 61% ethyl styrene) (2640 g), 2-ethylhexyl acrylate (4720 g), and hexanedioldiacrylate (640 g) is added a diglycerol monooleate emulsifier (480 g), ditallow dimethyl ammonium methyl sulfate (80g), and Tinuvin 765 (20 g). The diglycerol monooleate emulsifier (Grindsted Products; Brabrand, Denmark) comprises approximately 81% diglycerol monooleate, 1% other diglycerol monoesters, 3% polyols, and 15% other polyglycerol esters, imparts a minimum oil/water interfacial tension value of approximately 2.7 dyne/cm and has an oil/water critical aggregation concentration of approximately 2.8 wt%. After mixing, this combination of materials is allowed to settle overnight. No visible residue is formed and all of the mixture is withdrawn and used as the oil phase in a continuous process for forming a HIPE emulsion.

Separate streams of the oil phase (25°C) and water phase (53°-55°C) are fed to a dynamic mixing apparatus. Thorough mixing of the combined streams in the dynamic mixing apparatus is achieved by means of a pin impeller. The pin impeller comprises a cylindrical shaft of about 36.5 cm in length with a diameter of about 2.9 cm. The shaft holds 6 rows of pins, 3 rows having 33 pins and 3 rows having 34 pins, each of the three pins at each level disposed at an angle of 120° to each other, with the next level down disposed at 60° to its neighboring level with each level separated by .03 mm, each pin having a diameter of 0.5 cm extending outwardly from the central axis of the shaft to a length of 2.3 cm. The pin impeller is mounted in a cylindrical sleeve which forms the dynamic mixing apparatus, and the pins have a clearance of 1.5 mm from the walls of the cylindrical sleeve.

A minor portion of the effluent exiting the dynamic mixing apparatus is withdrawn and enters a recirculation zone, as shown in the Figure in co-pending U.S. Patent application Serial No. 08/716,510 (T. A. DesMarais), filed September 17, 1996 (herein incorporated by reference). The Waukesha pump in the recirculation zone returns the minor portion to the entry point of the oil and water phase flow streams to the dynamic mixing zone.

A spiral static mixer is mounted downstream from the dynamic mixing apparatus to provide back pressure in the dynamic mixing apparatus and to provide improved incorporation of components into the HIPE that is eventually formed. The static mixer (TAH Industries Model 100-812) has 12 elements with a 1 inch (2.5 cm) outside diameter. A hose is mounted downstream from the static mixer to facilitate delivery of the emulsion to the device used for curing. Optionally an additional static mixer is used to provide addition back pressure to keep the hose filled. The optional static mixer can be a 1 inch (2.5 cm) pipe, 12 element mixer (McMaster-Carr Model 3529K53).

The combined mixing and recirculation apparatus set-up is filled with oil phase and water phase at a ratio of 4 parts water to 1 part oil. The dynamic mixing apparatus is vented to allow air to escape while filling the apparatus completely. The flow rates during filling are 7.57 g/sec oil phase and 30.3 cm<sup>3</sup>/sec water phase.

Once the apparatus set-up is filled, agitation is begun in the dynamic mixer, with the impeller turning at 850 RPM and recirculation is begun at a rate of about 30 cm<sup>3</sup>/sec. The flow rate of the water phase is then steadily increased to a rate of 151.3 cm<sup>3</sup>/sec over a time period of about 1 min., and the oil phase flow rate is reduced to 2.52 g/sec over a time period of about 3 min. The recirculation rate is steadily increased to about 150 cm<sup>3</sup>/sec during the latter time period. The back pressure created by the dynamic zone and static mixers at this point is about 4.9 PSI (33.8 kPa), which represents the total pressure drop of the system. The Waukesha pump speed is then steadily decreased to a yield a recirculation rate of about 75 cm<sup>3</sup>/sec.

The HIPE flowing from the static mixer at this point is collected in a round polyethylene tub, 40 in. (102 cm) in diameter and 12.5 in (31.8cm) high, with removable sides, much like a springform pan used in cooking cakes. A pipe-like polyethylene insert 12.5 in (31.8cm) in diameter at its base is firmly affixed to the center of the base and is 12.5 in (31.8cm) high. The HIPE-containing tubs are kept in a room maintained at 65 °C. for 18 hours to bring about polymerization and form the foam.

The cured HIPE foam is removed from the curing tubs. The foam at this point has residual water phase (containing dissolved emulsifiers, electrolyte, initiator residues, and initiator) about 55-65 times (55-65X) the weight of polymerized monomers. The foam is sliced with a sharp reciprocating saw blade into sheets which are 0.2 inches (5.1 mm) in thickness. These sheets are then subjected to compression in a series of 2 porous nip rolls equipped with vacuum which gradually reduce the residual water phase content of the foam to about 3 times (3X) the weight of the polymerized material. At this point, the sheets are then resaturated with a 4% CaCl<sub>2</sub> solution at 60°C., are squeezed in a series of 3 porous nip rolls equipped with vacuum to a water phase content of about 1.5-2X. The CaCl<sub>2</sub> content of the foam is between 6 and 10 %.

The foam remains compressed after the final nip at a thickness of about 0.027 in. (0.069 cm). The foam is then dried in air for about 16 hours. Such

drying reduces the moisture content to about 9-17 % by weight of polymerized material. At this point, the foam sheets are very drapeable.

### Sample 2

Anhydrous calcium chloride (36.32 kg) and potassium persulfate (189 g) are dissolved in 378 liters of water. This provides the water phase stream to be used in a continuous process for forming a HIPE emulsion.

To a monomer combination comprising distilled divinylbenzene (42.4% divinylbenzene and 57.6% ethyl styrene) (2640 g), 2-ethylhexyl acrylate (4400 g), and hexanedioldiacrylate (960 g) is added a diglycerol monooleate emulsifier (640 g), ditallow dimethyl ammonium methyl sulfate (80g), and Tinuvin 765 (20 g). The diglycerol monooleate emulsifier (Grindsted Products; Brabrand, Denmark) comprises approximately 81% diglycerol monooleate, 1% other diglycerol monoesters, 3% polyols, and 15% other polyglycerol esters, imparts a minimum oil/water interfacial tension value of approximately 2.7 dyne/cm and has an oil/water critical aggregation concentration of approximately 2.8 wt%. After mixing, this combination of materials is allowed to settle overnight. No visible residue is formed and all of the mixture is withdrawn and used as the oil phase in a continuous process for forming a HIPE emulsion.

Separate streams of the oil phase (25°C) and water phase (75°-77°C) are fed to a dynamic mixing apparatus. Thorough mixing of the combined streams in the dynamic mixing apparatus is achieved by means of a pin impeller. The pin impeller comprises a cylindrical shaft of about 36.5 cm in length with a diameter of about 2.9 cm. The shaft holds 6 rows of pins, 3 rows having 33 pins and 3 rows having 34 pins, each of the three pins at each level disposed at an angle of 120° to each other, with the next level down disposed at 60° to its neighboring level with each level separated by .03 mm, each pin having a diameter of 0.5 cm extending outwardly from the central axis of the shaft to a length of 2.3 cm. The pin impeller is mounted in a cylindrical sleeve which forms the dynamic mixing apparatus, and the pins have a clearance of 1.5 mm from the walls of the cylindrical sleeve.

A minor portion of the effluent exiting the dynamic mixing apparatus is withdrawn and enters a recirculation zone, as shown in the Figure in co-pending U.S. Patent application Serial No. 08/716,510 (T. A. DesMarais), filed September 17, 1996 (herein incorporated by reference). The Waukesha pump in the recirculation zone returns the minor portion to the entry point of the oil and water phase flow streams to the dynamic mixing zone.

A spiral static mixer is mounted downstream from the dynamic mixing apparatus to provide back pressure in the dynamic mixing apparatus and to provide improved incorporation of components into the HIPE that is eventually formed. The static mixer (TAH Industries Model 101-212) normally has 12 elements with a 1.5 inch (3.8 cm) outside diameter, but 7 inches (17.8cm) were removed to fit in the equipment space. A hose is mounted downstream from the static mixer to facilitate delivery of the emulsion to the device used for curing. Optionally an additional static mixer is used to provide addition back pressure to keep the hose filled. The optional static mixer can be the same as the first without modification.

The combined mixing and recirculation apparatus set-up is filled with oil phase and water phase at a ratio of 4 parts water to 1 part oil. The dynamic mixing apparatus is vented to allow air to escape while filling the apparatus completely. The flow rates during filling are 7.57 g/sec oil phase and 30.3 cm<sup>3</sup>/sec water phase.

Once the apparatus set-up is filled, agitation is begun in the dynamic mixer, with the impeller turning at 800 RPM and recirculation is begun at a rate of about 30 cm<sup>3</sup>/sec. The flow rate of the water phase is then steadily increased to a rate of 151.3 cm<sup>3</sup>/sec over a time period of about 1 min., and the oil phase flow rate is reduced to 2.52 g/sec over a time period of about 3 min. The recirculation rate is steadily increased to about 150 cm<sup>3</sup>/sec during the latter time period. The back pressure created by the dynamic zone and static mixers at this point is about 4.2 PSI (29 kPa), which represents the total pressure drop of the system.

The HIPE flowing from the static mixer at this point is collected in a round polyethylene tub, 40 in. (102 cm) in diameter and 12.5 in (31.8cm) high, with removable sides, much like a springform pan used in cooking cakes. A pipe-like

polyethylene insert 12.5 in (31.8cm) in diameter at its base is firmly affixed to the center of the base and is 12.5 in (31.8cm) high. The HIPE-containing tubs are kept in a room maintained at 65 °C. for 18 hours to bring about polymerization and form the foam.

The cured HIPE foam is removed from the curing tubs. The foam at this point has residual water phase (containing dissolved emulsifiers, electrolyte, initiator residues, and initiator) about 58-62 times (58-62X) the weight of polymerized monomers. The foam is sliced with a sharp reciprocating saw blade into sheets which are 0.2 inches (5.1 mm) in thickness. These sheets are then subjected to compression in a series of 2 porous nip rolls equipped with vacuum which gradually reduce the residual water phase content of the foam to about 6 times (6X) the weight of the polymerized material. At this point, the sheets are then resaturated with a 1.5% CaCl<sub>2</sub> solution at 60°C., are squeezed in a series of 3 porous nip rolls equipped with vacuum to a water phase content of about 2X. The CaCl<sub>2</sub> content of the foam is between 3 and 6 %.

The foam remains compressed after the final nip at a thickness of about 0.047 in. (0.071 cm). The foam is then dried in air for about 16 hours. Such drying reduces the moisture content to about 9-17 % by weight of polymerized material. At this point, the foam sheets are very drapeable.

### Sample 3

Anhydrous calcium chloride (36.32 kg) and potassium persulfate (189 g) are dissolved in 378 liters of water. This provides the water phase stream to be used in a continuous process for forming a HIPE emulsion.

To a monomer combination comprising distilled divinylbenzene (42.4% divinylbenzene and 57.6% ethyl styrene) (2640 g), 2-ethylhexyl acrylate (4400 g), and hexanedioldiacrylate (960 g) is added a diglycerol monooleate emulsifier (640 g), ditallow dimethyl ammonium methyl sulfate (80g), and Tinuvin 765 (40 g). The diglycerol monooleate emulsifier (Grindsted Products; Brabrand, Denmark) comprises approximately 81% diglycerol monooleate, 1% other diglycerol monoesters, 3% polyols, and 15% other polyglycerol esters, imparts a minimum oil/water interfacial tension value of approximately 2.7 dyne/cm and has an oil/water critical aggregation concentration of approximately 2.8 wt%. After

mixing, this combination of materials is allowed to settle overnight. No visible residue is formed and all of the mixture is withdrawn and used as the oil phase in a continuous process for forming a HIPE emulsion.

Separate streams of the oil phase (25°C) and water phase (75°-77°C) are fed to a dynamic mixing apparatus. Thorough mixing of the combined streams in the dynamic mixing apparatus is achieved by means of a pin impeller. The pin impeller comprises a cylindrical shaft of about 21.6 cm in length with a diameter of about 1.9 cm. The shaft holds 6 rows of pins, one level with 3 rows having 21 pins and another level with 3 rows having 21 pins, each of the three pins at each level disposed at an angle of 120° to each other, with the next level down disposed at 60° to its neighboring level with each level separated by .03 mm, each having a diameter of 0.5 cm extending outwardly from the central axis of the shaft to a length of 1.4 cm. The pin impeller is mounted in a cylindrical sleeve which forms the dynamic mixing apparatus, and the pins have a clearance of 3 mm from the walls of the cylindrical sleeve.

A minor portion of the effluent exiting the dynamic mixing apparatus is withdrawn and enters a recirculation zone, as shown in the Figure in co-pending U.S. Patent application Serial No. 08/716,510 (T. A. DesMarais), filed September 17, 1996 (herein incorporated by reference). The Waukesha pump in the recirculation zone returns the minor portion to the entry point of the oil and water phase flow streams to the dynamic mixing zone.

A spiral static mixer is mounted downstream from the dynamic mixing apparatus to provide back pressure in the dynamic mixing apparatus and to provide improved incorporation of components into the HIPE that is eventually formed. The static mixer (TAH Industries Model 070-821), modified by cutting off 2.4 inches (6.1 cm) of its original length) is 14 inches (35.6 cm) long with a 0.5 inch (1.3 cm) outside diameter.

The combined mixing and recirculation apparatus set-up is filled with oil phase and water phase at a ratio of 4 parts water to 1 part oil. The dynamic mixing apparatus is vented to allow air to escape while filling the apparatus completely. The flow rates during filling are 1.89 g/sec oil phase and 7.56 cm<sup>3</sup>/sec water phase.



Once the apparatus set-up is filled, agitation is begun in the dynamic mixer, with the impeller turning at 1000 RPM and recirculation is begun at a rate of about 8 cm<sup>3</sup>/sec. The flow rate of the water phase is then steadily increased to a rate of 45.4 cm<sup>3</sup>/sec over a time period of about 1 min., and the oil phase flow rate is reduced to .6 g/sec over a time period of about 3 min. The recirculation rate is steadily increased to about 45 cm<sup>3</sup>/sec during the latter time period. The back pressure created by the dynamic zone and static mixers at this point is about 2.9 PSI (20 kPa), which represents the total pressure drop of the system.

The HIPE flowing from the static mixer at this point is collected in a round polypropylene tub, 17 in. (43 cm) in diameter and 7.5 in (10 cm) high, with a concentric insert made of Celcon plastic. The insert is 5 in (12.7 cm) in diameter at its base and 4.75 in (12 cm) in diameter at its top and is 6.75 in (17.1 cm) high. The HIPE-containing tubs are kept in a room maintained at 65 °C. for 18 hours to bring about polymerization and form the foam.

The cured HIPE foam is removed from the curing tubs. The foam at this point has residual water phase (containing dissolved emulsifiers, electrolyte, initiator residues, and initiator) about 70-80 times (70-80X) the weight of polymerized monomers. The foam is sliced with a sharp reciprocating saw blade into sheets which are 0.185 inches (4.7 mm) in thickness. These sheets are then subjected to compression in a series of 2 porous nip rolls equipped with vacuum which gradually reduce the residual water phase content of the foam to about 3 times (3X) the weight of the polymerized material. At this point, the sheets are then resaturated with a 1.5% CaCl<sub>2</sub> solution at 60°C., are squeezed in a series of 3 porous nip rolls equipped with vacuum to a water phase content of about 2X. The CaCl<sub>2</sub> content of the foam is between 3 and 5 %.

The foam remains compressed after the final nip at a thickness of about 0.031 in. (0.079 cm). The foam is then dried in air for about 16 hours. Such drying reduces the moisture content to about 9-17 % by weight of polymerized material. At this point, the foam sheets are very drapeable.

#### Test procedures

Unless specified otherwise, the tests are carried out under controlled laboratory conditions of about 23 +/- 2°C and at 50 +/-10% relative humidity. Test specimen are stored under these conditions for at least 24 hours before testing. It will be recognized that where the test material lacks sufficient integrity to withstand the testing protocol, a hydrophobic screen that does not impact wicking performance can be used to support the material

#### Synthetic urine formulation.

Unless specified explicitly, the specific synthetic urine used in the test methods is commonly known as Jayco SynUrine and is available from Jayco Pharmaceuticals Company of Camp Hill, Pennsylvania. The formula for the synthetic urine is: 2.0 g/l of KCl; 2.0 g/l of Na<sub>2</sub>SO<sub>4</sub>; 0.85 g/l of (NH<sub>4</sub>)H<sub>2</sub>PO<sub>4</sub>; 0.15 g/l (NH<sub>4</sub>)H<sub>2</sub>PO<sub>4</sub>; 0.19 g/l of CaCl<sub>2</sub>; ad 0.23 g/l of MgCl<sub>2</sub>. All of the chemicals are of reagent grade. The pH of the synthetic Urine is in the range of 6.0 to 6.4.

#### Vertical Wicking Time and Vertical Wicking Capacity

Vertical wicking time is determined by measuring the time taken for a colored test liquid (e.g., synthetic urine) in a reservoir to wick a vertical distance of 15 cm through a test strip of foam of specified size. The vertical wicking procedure is detailed in the Test Methods section of U.S. Patent No. 5,387,207 (which is incorporated by reference,) *supra*, but is performed at 31°C instead of 37°C. A material's vertical wicking capacity for a given height is measured using the Vertical Wicking Absorbent Capacity Test also described in the Test Methods section of U.S. Patent No. 5,387,207, except the test is performed at 31°C instead of 37°C. Finally, the washing and redrying step in the referenced patent is not performed. The vertical wicking capacity value of note is taken as the capacity achieved at a height of 15 cm at equilibrium. The result is expressed in units of (g/cm<sup>2</sup>/sec), at a height of 15 cm.

#### Vertical Wicking Flux Test

For the purpose of the present invention, it has been found, that a preferred method of determination of the vertical wicking properties can be achieved by using the Vertical Wicking Flux Test, which better characterises the ability of an absorbent material (distribution material or acquisition/distribution material) to wick liquid vertically. Any material that has enough dry and wet integrity to be hung vertically can be tested. If a material has not sufficient integrity, an

additional mechanical support layer can be added on either one or both surfaces of the material, such as low basis weight scrim or net, which does not or only negligibly impact on any fluid handling property.

In essence, this test is run by hanging 20 cm of a strip of the sample (in total at least 27.5 cm long) vertically in a reservoir of the test liquid, whereby the remaining 7.5 cm of the sample is placed horizontally on a plastic surface. Electrical conductivity sensors, are used to determine the time it takes to reach specific heights. Uptake vs. time data is obtained from the loss in weight of the liquid in the reservoir. At the end of the experiment, the sample is cut into sections to determine the longitudinal saturation profile.

Thereby, the VWF test gives the following information:

- Uptake (expressed in grams, or grams per cross-sectional area of the sample, or grams per weight of sample) vs. time
- Height of liquid front vs. time
- Flux through the sample (cumulative or incremental) vs. time
- Saturation profile along the length of the strip at the end of the experiment

The following equipment is utilized to execute the test (refer to Figure 7):

Motorized slide stand (950) (such as supplied by Concord-Renn Co., Cincinnati, Ohio, US) adjusted to obtain proper height of 20 cm from fluid reservoir (960) to horizontal laydown of sample (910). The motorized slide (950) ensures that the sample (910) is in contact with the liquid all the time by using a zero point sensor that maintains the apparatus at a position that is relative to the fluid level (962) in the reservoir (960). The slide stand (950) is further equipped with conductivity sensitive moisture detection rollers (930). A total of 9 rollers (930) are spaced individually at 2, 3.5, 5, 7.5, 10, 12.5, 15, 17.5, and 20 cm height above the level of the reservoir (962) (with the center of the rolls defining this measure). Two further sensors (930) are positioned in the horizontal part of the slide, so as to contact the sample (910) a running length of 22.5 cm and 27.5 cm from the liquid level. Although the exact width of the rollers (930) is insignificant, about 1/2" width has been found suitable, as well as a diameter of 3/8" (about 9.5 mm). It must be ensured, that the center of the roller (930) coincides with those heights. The rollers at 2, 5, 10, 15, and 20 cm are mounted to an arm (940) in a ladder-like fashion, and the rollers at 3.5, 7.5, 12.5, and 17.5 cm are mounted on another arm (940) in the same ladder-like fashion. During the

testing, these two arms (940) are positioned on the opposite surfaces of the testing material (910), thus also allowing testing of materials of different calipers. During testing, the arms (940) are arranged parallel, angled about 5° to the vertical.

The circular reservoir (960) can be glass bowl of high enough capacity that the fluid level does not drop significantly during the testing such as Pyrex #3140 bowl of dimensions 150 mm diameter by 75 mm height. The reservoir (960) is covered to reduce evaporation by a lid having a slit of about (1" X 2" or about 2.5 by 5 cm). The reservoir (960) is placed on a balance (965), such as Mettler PR 1203, Sartorius LC 1200S.

In order to ensure the required constant temperature and relative humidity conditions of 88°F (31.1 °C) and 85% RH - unless otherwise desired and defined, the total measurement equipment can be placed inside as Environmental chamber - such as Electro-Tech Systems, Model 518.

A personal computer system (980) is used to record - as a function of time - the weight change of the scale (965), the signal of the rollers (930) when these are reached by the liquid front and the height of the motorized slide stand (950). Whilst various programs can be used to achieve this, a suitable program has been developed by Signalysis, Inc., 431 Ohio Pike, Cincinnati, Ohio, US (see below for more details).

In order to determine the weight of the sample sections, a pair of scissors, a ruler, and an analytical balance such as Mettler PG503 are required.

The synthetic urine used herein is the same as described in the above.

#### Experimental Procedure

The test sample (910) is pre-conditioned overnight under standard conditions (72°F / 22.2 °C and 50% RH), and the environmental chamber to is set to 88°F / 31.1° C and 85% RH and equilibrated for approximately 45 min.

A 5.0 cm x 27.5 cm sample piece (910) is cut such as by a cutting die of those dimensions, and eleven 2.5 cm segments are marked by placing fine dots on the edges of the sample. The sample (910) is then pre-weighed, and its caliper is measured at about 0.09 psi (620 Pa) pressure by means of a standard caliper gauge.

The sample (910) is placed in between the rollers (930) such that the bottom of the sample is about 1 mm below the zero point on the VWF apparatus, without dipping in the liquid reservoir at this point. The top end of the sample (910) is fixed to the plastic plate such as by using a conventional adhesive tape (970).

The environmental chamber is closed, and equilibrated to the set temperature and RH, again for approximately 20 minutes.

The computerized unit (980) is initialized by input of the pertinent data into the program, e.g. sample name, desired test duration, sample length, width, caliper, temperature, RH). The test is started by immersing the sample via lowering the motorized slide into the test liquid and i) uptake vs. time and ii) times to reach specified heights are recorded continuously. The screen can display an uptake vs. time plot, while the experiment is in progress.

At the end of the experiment, the sample (910) is automatically raised from the reservoir (960), and the saturation profile is determined, whilst ensuring that the sample is in the same vertical position that it was in during the experiment. This is necessary to ensure that there is no redistribution of liquid inside the sample while being cut into sections. The sample is removed from the test stand held vertically while 2.5 cm strips are cut and placed in pre-weighed dishes. The strips have to be cut from the bottom up.

Then, the horizontal portion of the sample is also cut into 2.5 cm sections, each placed in pre-weighed plastic dishes. Then, the wet weight of the sections is immediately determined to minimize evaporation losses. Then, the sections are dried in the oven at 150 °F overnight and re-weighed to determine the dry weights.

CalculationsUptake

The uptake can be in the following units

Q: gm

Q\*: gm/cm<sup>2</sup> of initial dry cross-sectional area

Q': gm/gm of initial total sample weight

Qc': evaporation corrected uptake in gm/cm<sup>2</sup>

The evaporation loss from a partially covered reservoir (88°F /31.1°C and 85% RH) has to be monitored (e.g. about 0.009 g/min).

In addition to this, the evaporation loss of liquid from the wetted part of the sample has to be considered for long lasting experiments (e.g. more than about two hours).

The "Evaporation corrected Uptake" [in g/min] can be calculated by subtracting the cumulative evaporation loss (i.e. the reservoir evaporation [in units of g/min] multiplied with the time [in units of min]) from the weight of the fluid in the reservoir [in g], and dividing it by the sample width and caliper [both in cm].

When data is taken at n distinct times for  $i=1, \dots, n$  the following quantities are calculated:

Cumulative Flux,  $F_i$  (g/cm<sup>2</sup>/min)

$$F_i = Q_i^*/t_i$$

where  $Q_i^*$  is the uptake (g/cm<sup>2</sup>)

and  $t_i$  is the time (min)

Incremental Flux,  $\Delta F_i$  (g/cm<sup>2</sup>/min)

$$\Delta F_i = (Q_i^* - Q_{i-1}^*) / (t_i - t_{i-1})$$

### Saturation Profile Along the Length of the Sample

Liquid loading in each section (g/g) =  $(W_{\text{wet}} - W_{\text{dry}}) / W_{\text{dry}}$

where  $W_{\text{wet}}$  = weight of the wet section (g)

and  $W_{\text{dry}}$  = weight of the dry section

The following describes the Computer Programming Information in more detail: The Wicking Flux program is used to acquire fluid uptake rate data versus time for experiments run to quantify the absorbent characteristics of diaper materials. The program works by reading serial data from a balance equipped with an RS-232 output. The internal clock of the PC is used to track time. The program is written for an IBM PC or compatible with DOS 3.0 (or later) system.

Whenever an actual experiment is run, the data from the experiment (uptake values and time) and the values for all the input fields (except the sampling table) is written to a file in the current DOS directory. This file can be opened by Excel on an IBM station for further data analysis and presentation.

A typical test protocol is described below:

#### Input fields

Material description

Material identification

Sample #1

Operator: Fluid Type: Jayco synthetic urine

Test Duration: 60 minutes

Width: 5.0000 cm Length: 27.5 cm Weight: 1.9000 g

Temperature: 88.0000 °F Relative Humidity: 85.0000 %

#### Output

Time(min)	Weight(g)	Uptake(g/sq-cm)	Uptake(g/g)	Height(cm)
0	0.424011	0.77093	0.223164	2
0.04485	0.924011	1.68002	0.486322	3.5
0.0778167	1.22	2.21818	0.642106	5
0.247167	2.017	3.66727	1.06158	7.5
0.552	2.86401	5.2073	1.50738	10
1.12688	3.79501	6.90002	1.99738	12.5
1.87295	4.5	8.18182	2.36842	15
3.32845	5.409	9.83454	2.84684	17.5

5.53095                      6.25302                      11.3691                      3.29106                      20

### Reporting

At least 2 measurements should be made on each sample, recording the various out-put parameter as a function of time. Of course, the individual parameter can be correlated to each other, and also plotted accordingly.

For the purpose of describing materials suitable for being used within the present description, the cumulative flux at 15 cm height has been found to be particularly useful.

### Simplified Liquid Permeability Test

This Simplified Permeability Test provides a measure for permeability for two special conditions: Either the permeability can be measured for a wide range of porous materials (such as non-wovens made of synthetic fibres, or cellulosic structures) at 100% saturation, or for materials, which reach different degrees of saturation with a proportional change in caliper without being filled with air (respectively the outside vapour phase), such as the collapsible polymeric foams, for which the permeability at varying degrees of saturation can readily be measured at various thicknesses.

In particular for polymeric foam materials, it has been found useful to operate the test at an elevated temperature of 31°C, so as to better simulate in-use conditions for absorbent articles.

In principle, this tests is based on Darcy's law, according to which the volumetric flow rate of a liquid through any porous medium is proportional to the pressure gradient, with the proportionality constant related to permeability.

$$Q/A = (k/\eta) * (\Delta P/L)$$

where:

Q= Volumetric Flow Rate [cm<sup>3</sup>/s];

A= Cross Sectional Area [cm<sup>2</sup>];

k= Permeability (cm<sup>2</sup>) (with 1 Darcy corresponding to 9.869\* 10<sup>-13</sup> m<sup>2</sup>);

η= Viscosity (Poise) [Pa\*s];

ΔP/L= Pressure Gradient [Pa/m];

L= caliper of sample [cm].



Hence, permeability can be calculated - for a fixed or given sample cross-sectional area and test liquid viscosity - by measurement of pressure drop and the volumetric flow rate through the sample:

$$k = (Q/A) * (L/\Delta P) * \eta$$

The test can be executed in two modifications, the first referring to the transplanar permeability (i.e. the direction of flow is essentially along the thickness dimension of the material), the second being the in-plane permeability (i.e. the direction of flow being in the x-y-direction of the material).

The test set-up for the simplified, transplanar permeability test can be seen in Figure 1 which is a schematic diagram of the overall equipment and - as an insert diagram - a partly exploded cross-sectional, not to scale view of the sample cell.

The test set-up comprises a generally circular or cylindrical sample cell (120), having an upper (121) and lower (122) part. The distance of these parts can be measured and hence adjusted by means of each three circumferentially arranged caliper gauges (145) and adjustment screws (140). Further, the equipment comprises several fluid reservoirs (150, 154, 156) including a height adjustment (170) for the inlet reservoir (150) as well as tubings (180), quick release fittings (189) for connecting the sample cell with the rest of the equipment, further valves (182, 184, 186, 188). The differential pressure transducer (197) is connected via tubing (180) to the upper pressure detection point (194) and to the lower pressure detection point (196). A Computer device (190) for control of valves is further connected via connections (199) to differential pressure transducer (197), temperature probe (192), and weight scale load cell (198).

The circular sample (110) having a diameter of 1 in (about 2.54 cm) is placed in between two porous screens (135) inside the sample cell (120), which is made of two 1 in (2.54 cm) inner diameter cylindrical pieces (121, 122) attached via the inlet connection (132) to the inlet reservoir (150) and via the outlet connection (133) to the outlet reservoir (154) by flexible tubing (180), such as tygon tubing. Closed cell foam gaskets (115) provide leakage protection around the sides of the sample. The test sample (110) is compressed to the caliper corresponding to the desired wet compression, which is set to 0.2 psi (about 1.4 kPa) unless

otherwise mentioned. Liquid is allowed to flow through the sample (110) to achieve steady state flow. Once steady state flow through the sample (110) has been established, volumetric flow rate and pressure drop are recorded as a function of time using a load cell (198) and the differential pressure transducer (197). The experiment can be performed at any pressure head up to 80 cm water (about 7.8 kPa), which can be adjusted by the height adjusting device (170). From these measurements, the flow rate at different pressures for the sample can be determined.

The equipment is commercially available as a Permeameter such as supplied by Porous Materials, Inc, Ithaca, New York, US under the designation PMI Liquid Permeameter, such as further described in respective user manual of 2/97. This equipment includes two Stainless Steel Frits as porous screens (135), also specified in said brochure. The equipment consists of the sample cell (120), inlet reservoir (150), outlet reservoir (154), and waste reservoir (156) and respective filling and emptying valves and connections, an electronic scale, and a computerized monitoring and valve control unit (190).

The gasket material (115) is a Closed Cell Neoprene Sponge SNC-1 (Soft), such as supplied by Netherland Rubber Company, Cincinnati, Ohio, US. A set of materials with varying thickness in steps of 1/16" (about 0.159 cm) should be available to cover the range from 1/16" - 1/2" (about 0.159 cm to about 1.27 cm) thickness.

Further a pressurized air supply is required, of at least 60 psi (4.1 bar), to operate the respective valves.

Test fluid is deionized water.

The test is then executed by the following steps:

1) Preparation of the test sample(s):

In a preparatory test, it is determined, if one or more layers of the test sample are required, wherein the test as outlined below is run at the lowest and highest pressure. The number of layers is then adjusted so as to maintain the flow rate during the test between 0.5 cm<sup>3</sup>/seconds at the lowest pressure drop and 15 cm<sup>3</sup>/second at the highest pressure drop. The flow rate for the sample should be less than the flow rate for the blank at the same pressure drop. If the sample flow

rate exceeds that of the blank for a given pressure drop, more layers should be added to decrease the flow rate.

**Sample size:** Samples are cut to 1" (about 2.54 cm) diameter, by using an arch punch, such as supplied by McMaster-Carr Supply Company, Cleveland, OH, US. If samples have too little internal strength or integrity to maintain their structure during the required manipulation, a conventional low basis weight support means can be added, such as a PET scrim or net.

Thus, at least two samples (made of the required number of layers each, if necessary) are precut. Then, one of these is saturated in deionized water at the temperature the experiment is to be performed (70 ° F, (31 ° C) unless otherwise noted).

The caliper of the wet sample is measured (if necessary after a stabilization time of 30 seconds) under the desired compression pressure for which the experiment will be run by using a conventional caliper gauge (such as supplied by AMES, Waltham, MASS, US) having a pressure foot diameter of 1 1/8 " (about 2.86 cm), exerting a pressure of 0.2 psi (about 1.4 kPa) on the sample (110), unless otherwise desired.

An appropriate combination of gasket materials is chosen, such that the total thickness of the gasketing foam (115) is between 150 and 200% of the thickness of the wet sample (note that a combination of varying thicknesses of gasket material may be needed to achieve the overall desired thickness). The gasket material (115) is cut to a circular size of 3" in diameter, and a 1 inch (2.54 cm) hole is cut into the center by using the arch punch.

In case, that the sample dimensions change upon wetting, the sample should be cut such that the required diameter is taken in the wet stage. This can also be assessed in this preparatory test, with monitoring of the respective dimensions. If these change such that either a gap is formed, or the sample forms wrinkles which would prevent it from smoothly contacting the porous screens or frits, the cut diameter should be adjusted accordingly.

The test sample (110) is placed inside the hole in the gasket foam (115), and the composite is placed on top of the bottom half of the sample cell, ensuring that

the sample is in flat, smooth contact with the screen (135), and no gaps are formed at the sides.

The top of the test cell (121) is laid flat on the lab bench (or another horizontal plane) and all three caliper gauges (145) mounted thereon are zeroed.

The top of the test cell (121) is then placed onto the bottom part (122) such that the gasket material (115) with the test sample (110) lays in between the two parts. The top and bottom part are then tightened by the fixation screws (140), such that the three caliper gauges are adjusted to the same value as measured for the wet sample under the respective pressure in the above.

2) To prepare the experiment, the program on the computerized unit (190) is started and sample identification, respective pressure etc. are entered.

3) The test will be run on one sample (110) for several pressure cycles, with the first pressure being the lowest pressure. The results of the individual pressure runs are put on different result files by the computerized unit (190). Data are taken from each of these files for the calculations as described below. (A different sample should be used for any subsequent runs of the material.)

4) The inlet liquid reservoir (150) is set to the required height and the test is started on the computerized unit (190).

5) Then, the sample cell (120) is positioned into the permeameter unit with Quick Disconnect fittings (189).

6) The sample cell (120) is filled by opening the vent valve (188) and the bottom fill valves (184, 186). During this step, care must be taken to remove air bubbles from the system, which can be achieved by turning the sample cell vertically, forcing air bubbles - if present - to exit the permeameter through the drain.

Once the sample cell is filled up to the tygon tubing attached to the top of the chamber (121), air bubbles are removed from this tubing into the waste reservoir (156).

7) After having carefully removed air bubbles, the bottom fill valves (184, 186) are closed, and the top fill (182) valve is opened, so as to fill the upper part, also carefully removing all air bubbles.

8) The fluid reservoir is filled with test fluid to the fill line (152).

Then the flow is started through the sample by initiating the computerized unit (190).

After the temperature in the sample chamber has reached the required value, the experiment is ready to begin.

Upon starting the experiment via the computerized unit (190), the liquid outlet flow is automatically diverted from the waste reservoir (156) to the outlet reservoir (154), and pressure drop, and temperature are monitored as a function of time for several minutes.

Once the program has ended, the computerized unit provides the recorded data (in numeric and/or graphical form).

If desired, the same test sample can be used to measure the permeability at varying pressure heads, with thereby increasing the pressure from run to run.

The equipment should be cleaned every two weeks, and calibrated at least once per week, especially the frits, the load cell, the thermocouple and the pressure transducer, thereby following the instructions of the equipment supplier.

The differential pressure is recorded via the differential pressure transducer connected to the pressure probes measurement points (194, 196) in the top and bottom part of the sample cell. Since there may be other flow resistances within the chamber adding to the pressure that is recorded, each experiment must be corrected by a blank run. A blank run should be done at 10, 20, 30, 40, 50, 60, 70, 80 cm requested pressure, each day. The permeameter will output a Mean Test Pressure for each experiment and also an average flow rate.

For each pressure that the sample has been tested at, the flow rate is recorded as Blank Corrected Pressure by the computerized unit (190), which is

further correcting the Mean Test Pressure (Actual Pressure) at each height recorded pressure differentials to result in the Corrected Pressure. This Corrected Pressure is the DP that should be used in the permeability equation below.

Permeability can then be calculated at each requested pressure and all permeabilities should be averaged to determine the  $k$  for the material being tested.

Three measurements should be taken for each sample at each head and the results averaged and the standard deviation calculated. However, the same sample should be used, permeability measured at each head, and then a new sample should be used to do the second and third replicates.

The measuring of the in-plane permeability under the same conditions as the above described transplanar permeability, can be achieved by modifying the above equipment such as schematically depicted in Figures 2A and 2B showing the partly exploded, not to scale view of the sample cell only. Equivalent elements are denoted equivalently, such that the sample cell of Figure 2 is denoted (210), correlating to the numeral (110) of figure 1, and so on. Thus, the transplanar simplified sample cell (120) of figure 1 is replaced by the in-plane simplified sample cell (220), which is designed so that liquid can flow only in one direction (either machine direction or cross direction depending on how the sample is placed in the cell). Care should be taken to minimize channeling of liquid along the walls (wall effects), since this can erroneously give high permeability reading. The test procedure is then executed quite analogous to the transplanar simplified test.

The sample cell (220) is designed to be positioned into the equipment essentially as described for the sample cell (120) in the above transplanar test, except that the filling tube is directed to the inlet connection (232) the bottom of the cell (220). Figure 2A shows a partly exploded view of the sample cell, and Figure 2B a cross-sectional view through the sample level.

The test cell (220) is made up of two pieces: a bottom piece (225) which is like a rectangular box with flanges, and a top piece (223) that fits inside the bottom piece (225) and has flanges as well. The test sample is cut to the size of

2" in x 2" in (about 5.1 cm by 5.1 cm) and is placed into the bottom piece. The top piece (223) of the sample chamber is then placed into the bottom piece (225) and sits on the test sample (210). An incompressible neoprene rubber seal (224) is attached to the upper piece (223) to provide tight sealing. The test liquid flows from the inlet reservoir to the sample space via Tygon tubing and the inlet connection (232) further through the outlet connection (233) to the outlet reservoir. As in this test execution the temperature control of the fluid passing through the sample cell can be insufficient due to lower flow rates, the sample is kept at the desired test temperature by the heating device (226), whereby thermostated water is pumped through the heating chamber (227). The gap in the test cell is set at the caliper corresponding to the desired wet compression, normally 0.2 psi ( about 1.4 kPa). Shims (216) ranging in size from 0.1 mm to 20.0 mm are used to set the correct caliper, optionally using combinations of several shims.

At the start of the experiment, the test cell (220) is rotated 90° (sample is vertical) and the test liquid allowed to enter slowly from the bottom. This is necessary to ensure that all the air is driven out from the sample and the inlet/outlet connections (232/233). Next, the test cell (220) is rotated back to its original position so as to make the sample (210) horizontal. The subsequent procedure is the same as that described earlier for transplanar permeability, i.e. the inlet reservoir is placed at the desired height, the flow is allowed to equilibrate, and flow rate and pressure drop are measured. Permeability is calculated using Darcy's law. This procedure is repeated for higher pressures as well.

For samples that have very low permeability, it may be necessary to increase the driving pressure, such as by extending the height or by applying additional air pressure on the reservoir in order to get a measurable flow rate. In plane permeability can be measured independently in the machine and cross directions, depending on how the sample is placed in the test cell.

#### General Liquid Permeability Test

The generalized permeability test can measure permeability as a function of saturation for any porous material. The principle of the tests is similar to the one for the Simplified Test, with the essential difference being that the sample is loaded with a defined amount of air in addition to the liquid loading, resulting in a

fixed degree of saturation. This is achieved by the test arrangement as schematically depicted in Figure 3 showing the principles as well as the specifics for the General Transplanar Permeability, and in Figure 4, showing the differences for the General In-plane Permeability. Unreferenced numerals correspond to the respective numerals of Fig 1 (e.g., waste reservoir (356) corresponds to waste reservoir (156) etc.).

Therein, also the sample cell (320/420) is mounted with fixation (341, not shown in Fig. 4) on a height adjustment device (372), in addition to the inlet reservoir (350) being height adjustable by a means (370). This inlet reservoir defines a first height difference (357) relative to the outlet reservoir (354), which relates to the differential pressure  $\Delta p$  (which denotes the pressure differential for calculating the permeability). This inlet reservoir (350) defines a second height difference (359) relative to the sample height which relates to the differential pressure  $\Delta p(c)$ , which denotes the pressure differential linked to the saturation in the sample, whereby higher capillary suction typically correlates to lower saturation.

The experiment is started at low  $\Delta P_c$  (close to zero cm of water) at which the sample will be at 100% saturation. Liquid flows through the sample due to the applied pressure drop  $\Delta p(c)$  (inlet reservoir height - outlet reservoir height). At steady state, the uptake of liquid in the outlet reservoir is measured as a function of time. Permeability can be calculated from the pressure drop and the volumetric flow rate data using Darcy's law. The exact degree of saturation can be obtained from the weight of the wet sample after the test compared to the dry sample before the test.

In order to measure the permeability at saturation below 100%, a new test sample is first brought to 100% saturation as described in the paragraph above. Next, the sample is moved to a higher height (10 cm for example) and is allowed to equilibrate at that height. During this time, liquid continuously flows from the inlet to the outlet reservoir. The saturation in the sample will decrease with time. When steady state is reached, i.e. when the uptake versus time plot is linear, the flow rate, pressure drop and saturation are measured as described above. This procedure is repeated for several sample heights using new samples.



It may be necessary to increase the pressure drop between the inlet and outlet reservoirs as the saturation decreases in order to get a measurable flow rate. This is because, for most porous materials, permeability decreases steeply with decreasing saturation. It is necessary to ensure that the pressure drop between the inlet and outlet reservoirs is much smaller than the capillary suction.

It is necessary to use wide liquid reservoirs (352, 354) in order to ensure that the liquid level does not change significantly while waiting for steady state to be reached.

This test gives permeability versus saturation for the desorption cycle, that is the sample has higher saturation to start with. Whilst of course permeability data can be generated for the absorption cycle, these should not be used in present evaluations, as some hysteresis effects might occur.

The sample cell (320) for the general transplanar permeability test differs from sample cell (120) of the simplified transplanar permeability test essentially in that it comprises two frits (335) arranged on top and underneath the sample (310). For the frits (335) it is necessary to ensure that most of the resistance to flow is offered by the sample and the frit resistance is negligible. A fine pored, thin membrane over a coarse frit allows measurements up to high heights without offering significant resistance to flow. The frits should be selected so as to have a sufficiently high bubble point pressure corresponding to more than about 200 cm water height, but at the same time providing low flow resistance. This can well be achieved by selecting sufficiently thin membranes of the required bubble point pressure overlying a more open support structure.

For the general permeability tests, care must be taken, that the air is allowed to contact the sample via the side surfaces, so as to allow varying degrees of saturation depending on the  $\Delta p(c)$ . Thus, the sample cell design is essentially identical to the test cell of the simplified transplanar test, except, that the foam gasketing material is removed, and the arrangement to adjust the gap between the top and the bottom parts replaced by a constant pressure generating device, such as a weight (317) to maintain (together with the weight of the top piece (321)) the sample under the desired pressure, of 0.2 psi (about 1.4 kPa) unless otherwise desired.

For the general in-plane permeability test the sample cell (420) is shown in Figure 4, which is a design being derived from the simplified in-plane test and the principles as described in the above. Thus, the fluid is entering the sample cell (420) via the fluid inlet (432) and outlet (433), which are connected to the membranes (435), such as frits of the type as described above (for frits 335). The test sample (410) is positioned with its ends overlaying the two frits, but not with the center part of 2 in by 2 in (about 5.1 cm by 5.1 cm) whereby wrinkles and gaps between the sample and the membranes have to be avoided. The test sample (410) is placed between the upper and lower part of the sample cell (420), with the weight (417) being used to adjust the pressure under which the experiment is run (0.2 psi (about 1.4 kPa) unless otherwise desired and denoted). Also, the sample is kept at a constant temperature via the heating device (426), e.g. by pumping constant temperature water through the heating chamber (427).

Also for this set up, the possibility of air entering into the sample via the side surfaces is essential to allow the varying degrees of saturation.

#### Liquid Viscosity

The liquid viscosity is an important input parameter for the above determination, and should be taken for the respective fluid for the respective temperature, either from well known tables, or equations, or measured via well established measurement procedures.

#### Capillary Sorption

##### Purpose

The purpose of this test is to measure the capillary sorption absorbent capacity, as a function of height, of storage absorbent members of the present invention. (The test is also used to measure the capillary sorption absorbent capacity, as a function of height, of the high surface area materials - i.e., without osmotic absorbent, such as hydrogel-forming absorbent polymer, or other optional materials utilized in the absorbent member. Nonetheless, the discussion that follows discusses the Capillary Sorption method as it pertains to measuring an entire storage absorbent member.) Capillary sorption is a fundamental property of any absorbent

that governs how liquid is absorbed into the absorbent structure. In the Capillary Sorption experiment, capillary sorption absorbent capacity is measured as a function of fluid pressure due to the height of the sample relative to the test fluid reservoir.

The method for determining capillary sorption is well recognized. See Burgeni, A.A. and Kapur, C., "Capillary Sorption Equilibria in Fiber Masses," Textile Research Journal, 37 (1967), 356-366; Chatterjee, P.K., Absorbency, Textile Science and Technology 7, Chapter II, pp 29-84, Elsevier Science Publishers B.V, 1985; and U.S. Patent No. 4,610,678, issued September 9, 1986 to Weisman et al. for a discussion of the method for measuring capillary sorption of absorbent structures. The disclosure of each of these references is incorporated by reference herein.

#### Principle

A porous glass frit is connected via an uninterrupted column of fluid to a fluid reservoir on a balance. The sample is maintained under a constant confining weight during the experiment. As the porous structure absorbs fluid upon demand, the weight loss in the balance fluid reservoir is recorded as fluid uptake, adjusted for uptake of the glass frit as a function of height and evaporation. The uptake or capacity at various capillary suctions (hydrostatic tensions or heights) is measured. Incremental absorption occurs due to the incremental lowering of the frit (i.e., decreasing capillary suction).

Time is also monitored during the experiment to enable calculation of initial effective uptake rate (g/g/h) at a 200 cm height.

#### Reagents

Test Liquid: Synthetic urine is prepared by completely dissolving the following materials in distilled water.

<u>Compound</u>	<u>F.W.</u>	<u>Concentration (g/L)</u>
KCl	74.6	2.0

Na <sub>2</sub> SO <sub>4</sub>	142	2.0
(NH <sub>4</sub> )H <sub>2</sub> PO <sub>4</sub>	115	0.85
(NH <sub>4</sub> ) <sub>2</sub> HPO <sub>4</sub>	132	0.15
CaCl <sub>2</sub> ·2H <sub>2</sub> O	147	0.25
MgCl <sub>2</sub> ·6H <sub>2</sub> O	203	0.5

#### General Description of Apparatus Set Up

The Capillary Sorption equipment, depicted generally as **520** in Figure 5A, used for this test is operated under TAPPI conditions (50% RH, 25°C). A test sample is placed on a glass frit shown in Figure 5A as **502** that is connected via a continuous column of test liquid (synthetic urine) to a balance liquid reservoir, shown as **506**, containing test liquid. This reservoir **506** is placed on a balance **507** that is interfaced with a computer (not shown). The balance should be capable of reading to 0.001 g; such a balance is available from Mettler Toledo as PR1203 (Hightstown, NJ). The glass frit **502** is placed on a vertical slide, shown generally in Figure 5A as **501**, to allow vertical movement of the test sample to expose the test sample to varying suction heights. The vertical slide may be a rodless actuator which is attached to a computer to record suction heights and corresponding times for measuring liquid uptake by the test sample. A preferred rodless actuator is available from Industrial Devices (Novato, CA) as item 202X4X34N-1D4B-84-P-C-S-E, which may be powered by motor drive ZETA 6104-83-135, available from CompuMotor (Rohnert, CA). Where data is measured and sent from actuator **501** and balance **507**, capillary sorption absorbent capacity data may be readily generated for each test sample. Also, computer interface to actuator **501** may allow for controlled vertical movement of the glass frit **502**. For example, the actuator may be directed to move the glass frit **502** vertically only after "equilibrium" (as defined below) is reached at each suction height.

The bottom of glass frit **502** is connected to Tygon® tubing **503** that connects the frit **502** to three-way drain stopcock **509**. Drain stopcock **509** is connected to liquid reservoir **505** via glass tubing **504** and stopcock **510**. (The stopcock **509** is open to the drain only during cleaning of the apparatus or air bubble removal.) Glass tubing **511** connects fluid reservoir

505 with balance fluid reservoir 506, via stopcock 510. Balance liquid reservoir 506 consists of a lightweight 12 cm diameter glass dish 506A and cover 506B. The cover 506B has a hole through which glass tubing 511 contacts the liquid in the reservoir 506. The glass tubing 511 must not contact the cover 506B or an unstable balance reading will result and the test sample measurement cannot be used.

The glass frit diameter must be sufficient to accommodate the piston/cylinder apparatus, discussed below, for holding the test sample. The glass frit 502 is jacketed to allow for a constant temperature control from a heating bath. The frit is a 350 ml fritted disc funnel specified as having 4 to 5.5  $\mu\text{m}$  pores, available from Corning Glass Co. (Corning, NY) as #36060-350F. The pores are fine enough to keep the frit surface wetted at capillary suction heights specified (the glass frit does not allow air to enter the continuous column of test liquid below the glass frit).

As indicated, the frit 502 is connected via tubing to fluid reservoir 505 or balance liquid reservoir 506, depending on the position of three-way stopcock 510.

Glass frit 502 is jacketed to accept water from a constant temperature bath. This will ensure that the temperature of the glass frit is kept at a constant temperature of 88°F (31°C) during the testing procedure. As is depicted in Figure 5A, the glass frit 502 is equipped with an inlet port 502A and outlet port 502B, which make a closed loop with a circulating heat bath shown generally as 508. (The glass jacketing is not depicted in Figure 5A. However, the water introduced to the jacketed glass frit 502 from bath 508 does not contact the test liquid and the test liquid is not circulated through the constant temperature bath. The water in the constant temperature bath circulates through the jacketed walls of the glass frit 502.)

Reservoir 506 and balance 507 are enclosed in a box to minimize evaporation of test liquid from the balance reservoir and to enhance balance stability during performance of the experiment. This box, shown generally as 512, has a top and walls, where the top has a hole through which tubing 511 is inserted.

The glass frit **502** is shown in more detail in Figure 5B. Figure 5B is a cross-sectional view of the glass frit, shown without inlet port **502A** and outlet port **502B**. As indicated, the glass frit is a 350 ml fritted disc funnel having specified 4 to 5.5  $\mu\text{m}$  pores. Referring to Figure 5B, the glass frit **502** comprises a cylindrical jacketed funnel designated as **550** and a glass frit disc shown as **560**. The glass frit **502** further comprises a cylinder/piston assembly shown generally as **565** (which comprises cylinder **566** and piston **568**), which confines the test sample, shown as **570**, and provides a small confining pressure to the test sample. To prevent excessive evaporation of test liquid from the glass frit disc **560**, a Teflon ring shown as **562** is placed on top of the glass frit disc **560**. The Teflon® ring **562** is 0.0127 cm thick (available as sheet stock from McMasterCarr as # 8569K16 and is cut to size) and is used to cover the frit disc surface outside of the cylinder **566**, and thus minimizes evaporation from the glass frit. The ring outer diameter and inner diameter is 7.6 and 6.3 cm, respectively. The inner diameter of the Teflon® ring **562** is about 2 mm less than the outer diameter of cylinder **566**. A Viton® O-ring (available from McMasterCarr as # AS568A-150 and AS568A-151) **564** is placed on top of Teflon® ring **562** to seal the space between the inner wall of cylindrical jacketed funnel **550** and Teflon® ring **562**, to further assist in prevention of evaporation. If the O-ring outer diameter exceeds the inner diameter of cylindrical jacketed funnel **550**, the O-ring diameter is reduced to fit the funnel as follows: the O-ring is cut open, the necessary amount of O-ring material is cut off, and the O-ring is glued back together such that the O-ring contacts the inner wall of the cylindrical jacketed funnel **550** all around its periphery.

As indicated, a cylinder/piston assembly shown generally in Figure 5B as **565** confines the test sample and provides a small confining pressure to the test sample **570**. Referring to Figure 5C, assembly **565** consists of a cylinder **566**, a cup-like Teflon® piston indicated by **568** and, when necessary, a weight or weights (not shown) that fits inside piston **568**. (Optional weight will be used when necessary to adjust the combined weight of the piston and the optional weight so a confining pressure of 0.2 psi is attained depending on the test sample's dry diameter. This is discussed below.) The cylinder **566** is Lexan® bar stock and has the

following dimensions: an outer diameter of 7.0 cm, an inner diameter of 6.0 cm and a height of 6.0 cm. The Teflon® piston 568 has the following dimensions: an outer diameter that is 0.02 cm less than the inner diameter of cylinder 566. As shown in Figure 5D, the end of the piston 568 that does not contact the test sample is bored to provide a 5.0 cm diameter by about 1.8 cm deep chamber 590 to receive optional weights (dictated by the test sample's actual dry diameter) required to attain a test sample confining pressure of 0.2 psi (1.4 kPa). In other words, the total weight of the piston 568 and any optional weights (not shown in figures) divided by the test sample's actual diameter (when dry) should be such that a confining pressure of 0.2 psi is attained. Cylinder 566 and piston 568 (and optional weights) are equilibrated at 31°C for at least 30 minutes prior to conducting the capillary sorption absorbent capacity measurement.

A non-surfactant treated or incorporated apertured film (14 cm x 14 cm) (not shown) is used to cover the glass frit 502 during Capillary Sorption experiments to minimize air destabilization around the sample. Apertures are large enough to prevent condensation from forming on the underside of the film during the experiment.

#### Test Sample Preparation

The test sample can be obtained by punching out a 5.4 cm diameter circular-shaped structure from a storage absorbent member. When the member is a component of an absorbent article, other components of the article must be removed prior to testing. In those situations where the member cannot be isolated from other components of the article without significantly altering its structure (e.g., density, relative disposition of the component materials, physical properties of constituent materials, etc.) or where the member is not a component of an absorbent article, the test sample is prepared by combining all the materials that constitute the member such that the combination is representative of the member in question. The test sample is a 5.4 cm diameter circle and is obtained by cutting with an arch punch.

The dry weight of the test sample (used below to calculate capillary sorption absorbent capacity) is the weight of the test sample prepared as above under ambient conditions.

#### Experimental Set Up

1. Place a clean, dry glass frit **502** in a funnel holder attached to the vertical slide **501**. Move the funnel holder of the vertical slide such that the glass frit is at the 0 cm height.
2. Set up the apparatus components as shown in Figure 5A, as discussed above.
3. Place 12 cm diameter balance liquid reservoir **506** on the balance **507**. Place plastic lid **506B** over this balance liquid reservoir **506** and a plastic lid over the balance box **512** each with small holes to allow the glass tubing **511** to fit through. Do not allow the glass tubing to touch the lid **506B** of the balance liquid reservoir or an unstable balance reading will result and the measurement cannot be used.
4. Stopcock **510** is closed to tubing **504** and opened to glass tubing **511**. Fluid reservoir **505**, previously filled with test fluid, is opened to allow test fluid to enter tubing **511**, to fill balance fluid reservoir **506**.
5. The glass frit **502** is leveled and secured in place. Also, ensure that the glass frit is dry.
6. Attach the Tygon® tubing **503** to stopcock **509**. (The tubing should be long enough to reach the glass frit **502** at its highest point of 200 cm with no kinks.) Fill this Tygon® tubing with test liquid from liquid reservoir **505**.
7. Attach the Tygon® tubing **503** to the level glass frit **502** and then open stopcock **509** and stopcock **510** leading from fluid reservoir **505** to the glass frit **502**. (Stopcock **510** should be closed to glass tubing **511**.) The test liquid fills the glass frit **502** and removes all trapped air during filling of the level glass frit. Continue to fill until the fluid level exceeds the top of the glass frit disc **560**. Empty the funnel and remove all air bubbles in the



tubing and inside the funnel. Air bubbles may be removed by inverting glass frit **502** and allowing air bubbles to rise and escape through the drain of stopcock **509**. (Air bubbles typically collect on the bottom of the glass frit disc **560**.) Relevel the frit using a small enough level that it will fit inside the jacketed funnel **550** and onto the surface of glass frit disc **560**.

8. Zero the glass frit with the balance liquid reservoir **506**. To do this, take a piece of Tygon® tubing of sufficient length and fill it with the test liquid. Place one end in the balance liquid reservoir **506** and use the other end to position the glass frit **502**. The test liquid level indicated by the tubing (which is equivalent to the balance liquid reservoir level) is 10 mm below the top of the glass frit disc **560**. If this is not the case, either adjust the amount of liquid in the reservoir or reset the zero position on the vertical slide **501**.
9. Attach the outlet and inlet ports from the temperature bath **508** via tubing to the inlet and outlet ports **502A** and **502B**, respectively, of the glass frit. Allow the temperature of the glass frit disc **560** to come to 31°C. This can be measured by partially filling the glass frit with test liquid and measuring its temperature after it has reached equilibrium temperature. The bath will need to be set a bit higher than 31°C to allow for the dissipation of heat during the travel of water from the bath to the glass frit.
10. The glass frit is equilibrated for 30 minutes.

#### Capillary Sorption Parameters

The following describes a computer program that will determine how long the glass frit remains at each height.

In the capillary sorption software program, a test sample is at some specified height from the reservoir of fluid. As indicated above, the fluid reservoir is on a balance, such that a computer can read the balance at the end of a known time interval and calculate the flow rate (Delta reading/time interval) between the test sample and reservoir. For purposes of this

method, the test sample is considered to be at equilibrium when the flow rate is less than a specified flow rate for a specified number of consecutive time intervals. It is recognized, that for certain material, actual equilibrium may not be reached when the specified "EQUILIBRIUM CONSTANT" is reached. The time interval between readings is 5 seconds.

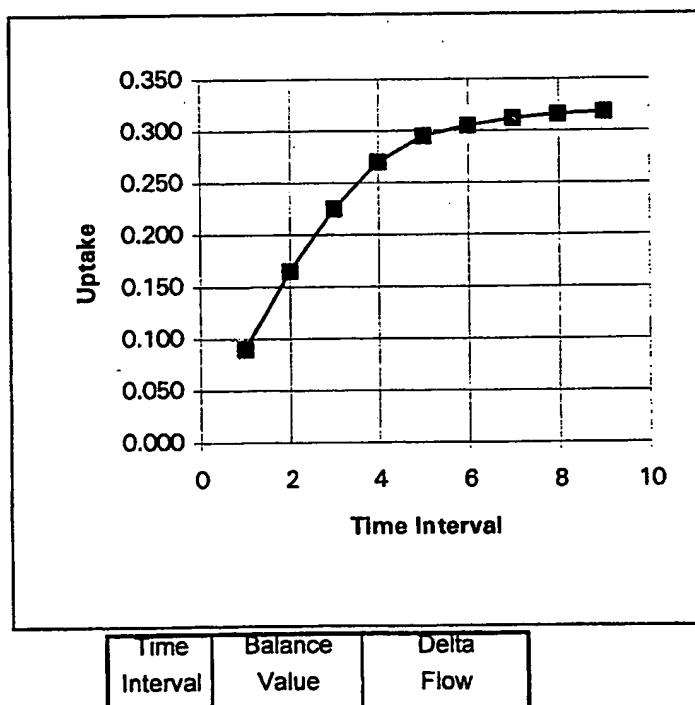
The number of readings in the delta table is specified in the capillary sorption menu as "EQUILIBRIUM SAMPLES". The maximum number of deltas is 500. The flow rate constant is specified in the capillary sorption menu as "EQUILIBRIUM CONSTANT".

The Equilibrium Constant is entered in units of grams/sec, ranging from 0.0001 to 100.000.

The following is a simplified example of the logic. The table shows the balance reading and Delta Flow calculated for each Time Interval.

Equilibrium Samples = 3

Equilibrium Constant = .0015



	(g)	(g/sec)
0	0	
1	0.090	0.0180
2	0.165	0.0150
3	0.225	0.0120
4	0.270	0.0090
5	0.295	0.0050
6	0.305	0.0020
7	0.312	0.0014
8	0.316	0.0008
9	0.318	0.0004

Delta Table:

Time	0	1	2	3	4	5	6	7	8	9
Delta1	9999	0.0180	0.0180	0.0180	0.0090	0.0090	0.0090	0.0014	0.0014	0.0014
Delta2	9999	9999	0.0150	0.0150	0.0150	0.0050	0.0050	0.0050	0.0008	0.0008
Delta3	9999	9999	9999	0.0120	0.0120	0.0120	0.0020	0.0020	0.0020	0.0004

The equilibrium uptake for the above simplified example is 0.318 gram.

The following is the code in C language used to determine equilibrium uptake:

```

/*                                takedata.c                                */
int take_data(int equil_samples, double equilibrium_constant)
{
    double    delta;
    static    double deltas[500]; /* table to store up to 500 deltas */
    double    value;
    double    prev_value;
    clock_t   next_time;
    int       i;

    for (i=0; i<equil_samples; i++)
        deltas[i] = 9999.; /* initialize all values in the delta */
    table_to_9999_gms/sec /* initialize where in the table to store */
    delta_table_index = 0; /* initialize flag to indicate equilibrium */
    the next delta /* initialize when to take the next */
    equilibrium_reached = 0; /* initialize the value of the previous */
    has not been reached /*
    next_time = clock();
    reading /*
    prev reading = 0.;
    reading from the balance */

    while (!equilibrium_reached) { /* start of loop for checking for
        equilibrium /*
            next_time += 5000L; /* calculate when to take next reading
        /* while (clock() < next_time); /* wait until 5 seconds has elapsed
        from prev reading */

```

```

    value = get_balance_reading();          /* read the balance in grams */
    delta = fabs(prev_value - value) / 5.0; /* calculate absolute value of flow in
last 5 seconds */
    prev_value = value;                    /* store current value for next loop
*/
    deltas[delta_table_index] = delta;      /* store current delta value in the
table of deltas */
    delta_table_index++;                   /* increment pointer to next position
in table */
    if (delta_table_index == equil_samples) /* when the number of deltas = the
number of */
        delta_table_index = 0;            /* equilibrium samples specified, /*
                                           /* reset the pointer to the start of
the table. This way */
                                           /* the table always contains the last
xx current samples. */
    equilibrium_reached = 1;               /* set the flag to indicate
equilibrium is reached */
    for (i=0; i < equil_samples; i++)      /* check all the values in the delta
table */
        if (deltas[i] >= equilibrium_constant) /* if any value is > or = to the
equilibrium constant */
            equilibrium_reached = 0;       /* set the equilibrium flag to 0 (not
at equilibrium) */
                                           /* go back to the start of the loop */
}

```

### Capillary Sorption Parameters

Load Description (Confining Pressure): 0.2 psi load

Equilibrium Samples (n): 50

Equilibrium Constant: 0.0005 g/sec

Setup Height Value: 100 cm

Finish Height Value: 0 cm

Hydrostatic Head Parameters: 200, 180, 160, 140, 120, 100, 90, 80,  
70, 60, 50, 45, 40, 35, 30, 25, 20, 15,  
10, 5 and 0 cm.

The capillary sorption procedure is conducted using all the heights specified above, in the order stated, for the measurement of capillary sorption absorbent capacity. Even if it is desired to determine capillary sorption absorbent capacity at a particular height (e.g., 35 cm), the entire series of hydrostatic head parameters must be completed in the order specified. Although all these heights are used in performance of the capillary sorption test to generate capillary sorption isotherms for a test sample, the present disclosure describes the storage absorbent members in terms of their absorbent properties at specified heights of 200, 140, 100, 50, 35 and 0 cm.

### Capillary Sorption Procedure

- 1) Follow the experimental setup procedure.
- 2) Make sure the temperature bath **508** is on and water is circulating through the glass frit **502** and that the glass frit disc **560** temperature is 31°C.
- 3) Position glass frit **502** at 200 cm suction height. Open stopcocks **509** and **510** to connect glass frit **502** with the balance liquid reservoir **506**. (Stopcock **510** is closed to liquid reservoir **505**.) Glass frit **502** is equilibrated for 30 minutes.
- 4) Input the above capillary sorption parameters into the computer.
- 5) Close stopcocks **509** and **510**.
- 6) Move glass frit **502** to the set up height, 100 cm.
- 7) Place Teflon® ring **562** on surface of glass frit disc **560**. Put O-ring **564** on Teflon® ring. Place pre-heated cylinder **566** concentrically on the Teflon® ring. Place test sample **570** concentrically in cylinder **566** on glass frit disc **560**. Place piston **568** into cylinder **566**. Additional confining weights are placed into piston chamber **590**, if required.
- 8) Cover the glass frit **502** with apertured film.
- 9) The balance reading at this point establishes the zero or tare reading.
- 10) Move the glass frit **502** to 200 cm.
- 11) Open stopcocks **509** and **510** (stopcock **510** is closed to fluid reservoir **505**) and begin balance and time readings.

### Glass Frit Correction (blank correct uptake)

Since the glass frit disc **560** is a porous structure, the glass frit (**502**) capillary sorption absorption uptake (blank correct uptake) must be determined and subtracted to get the true test sample capillary sorption absorption uptake. The glass frit correction is performed for each new glass frit used. Run the capillary sorption procedure as described above, except without test sample, to obtain the Blank Uptake (g). The elapsed time at each specified height equals the Blank Time (s).

### Evaporation Loss Correction

- 1) Move the glass frit **502** to 2 cm above zero and let it equilibrate at this height for 30 minutes with open stopcocks **509** and **510** (closed to reservoir **505**).
- 2) Close stopcocks **509** and **510**.
- 3) Place Teflon® ring **562** on surface of glass frit disc **560**. Put O-ring **564** on Teflon® ring. Place pre-heated cylinder **566** concentrically on the Teflon® ring. Place piston **568** into cylinder **566**. Place apertured film on glass frit **502**.
- 4) Open stopcocks **509** and **510** (closed to reservoir **505**) and record balance reading and time for 3.5 hours. Calculate Sample Evaporation (g/hr) as follows:

$$[\text{balance reading at 1 hr} - \text{balance reading at 3.5 hr}] / 2.5 \text{ hr}$$

Even after taking all the above precautions, some evaporative loss will occur, typically around 0.10 gm/hr for both the test sample and the frit correction. Ideally, the sample evaporation is measured for each newly installed glass frit **502**.

### Cleaning the Equipment

New Tygon® tubing **503** is used when a glass frit **502** is newly installed. Glass tubing **504** and **511**, fluid reservoir **505**, and balance liquid reservoir **506** are cleaned with 50% Clorox Bleach® in distilled water, followed by distilled water rinse, if microbial contamination is visible.

#### a. Cleaning after each experiment

At the end of each experiment (after the test sample has been removed), the glass frit is forward flushed (i.e., test liquid is introduced into the bottom of the glass frit) with 250 ml test liquid from liquid reservoir **505** to remove residual test sample from the glass frit disc pores. With stopcocks **509** and **510** open to liquid reservoir **505** and closed to balance liquid reservoir **506**, the glass frit is removed from its holder, turned upside

down and is rinsed out first with test liquid, followed by rinses with acetone and test liquid (synthetic urine). During rinsing, the glass frit must be tilted upside down and rinse fluid is squirted onto the test sample contacting surface of the glass frit disc. After rinsing, the glass frit is forward flushed a second time with 250 ml test liquid (synthetic urine). Finally, the glass frit is reinstalled in its holder and the frit surface is leveled.

b. Monitoring glass frit performance

Glass frit performance must be monitored after each cleaning procedure and for each newly installed glass frit, with the glass frit set up at 0 cm position. 50 ml of test liquid are poured onto the leveled glass frit disc surface (without Teflon® ring, O-ring and the cylinder/piston components). The time it takes for the test fluid level to drop to 5 mm above the glass frit disc surface is recorded. A periodic cleaning must be performed if this time exceeds 4.5 minutes.

c. Periodic cleaning

Periodically, (see monitoring frit performance, above) the glass frits are cleaned thoroughly to prevent clogging. Rinsing fluids are distilled water, acetone, 50% Clorox Bleach® in distilled water (to remove bacterial growth) and test liquid. Cleaning involves removing the glass frit from the holder and disconnecting all tubing. The glass frit is forward flushed (i.e., rinse liquid is introduced into the bottom of the glass frit) with the frit upside down with the appropriate fluids and amounts in the following order:

1. 250 ml distilled water.
2. 100 ml acetone.
3. 250 ml distilled water.
4. 100 ml 50:50 Clorox®/distilled water solution.
5. 250 ml distilled water.
6. 250 ml test fluid.

The cleaning procedure is satisfactory when glass frit performance is within the set criteria of fluid flow (see above) and when no residue is observable on the glass frit disc surface. If cleaning can not be performed successfully, the frit must be replaced.

### Calculations

The computer is set up to provide a report consisting of the capillary suction height in cm, time, and the uptake in grams at each specified height. From this data, the capillary suction absorbent capacity, which is corrected for both the frit uptake and the evaporation loss, can be calculated. Also, based on the capillary suction absorbent capacity at 0 cm, the capillary absorption efficiency can be calculated at the specified heights. In addition, the initial effective uptake rate at 200 cm is calculated.

#### Blank Correct Uptake

$$\text{Blank Correct Uptake (g)} = \text{Blank Uptake(g)} - \frac{\text{Blank Time(s)} * \text{Sample Evap. (g / hr)}}{3600(\text{s / hr})}$$

#### Capillary Suction Absorbent Capacity ("CSAC")

$$\text{CSAC(g/g)} = \frac{\text{Sample Uptake(g)} - \frac{\text{Sample Time(s)} * \text{Sample Evap. (g/hr)}}{3600 \text{ s/hr}} - \text{Blank Correct Uptake(g)}}{\text{Dry Weight of Sample(g)}}$$

#### Initial Effective Uptake Rate at 200 cm ("IEUR")

$$\text{IEUR (g/g/hr)} = \frac{\text{CSAC at 200 cm (g/g)}}{\text{Sample Time at 200 cm (s)}}$$

### Reporting

A minimum of two measurements should be taken for each sample and the uptake averaged at each height to calculate Capillary Sorption Absorbent Capacity (CSAC) for a given absorbent member or a given high surface area material.



With these data, the respective values can be calculated:

- The Capillary Sorption Desorption Height at which the material has released x% of its capacity at 0 cm (i.e. of CSAC 0), (CSDH x) expressed in cm;
- The Capillary Sorption Absorption Height at which the material has absorbed y % of its capacity at 0 cm (i.e. of CSAC 0), (CSAH y) expressed in cm;
- The Capillary Sorption Absorbent Capacity at a certain height z (CSAC z) expressed in units of g {of fluid} / g { of material}; especially at the height zero (CSAC 0), and at heights of 35cm, 40cm, etc
- The Capillary Sorption Absorption Efficiency at a certain height z (CSAE z) expressed in %, which is the ratio of the values for CSAC 0 and CSAC z.

If two materials are combined (such as the first being used as acquisition / distribution material, and the second being used as liquid storage material), the CSAC value (and hence the respective CSAE value) of the second material can be determined for the CSDH x value of the first material .

#### Teabag Centrifuge Capacity Test (TCC test)

Whilst the TCC test has been developed specifically for superabsorbent materials, it can readily be applied to other absorbent materials.

The Teabag Centrifuge Capacity test measures the Teabag Centrifuge Capacity values, which are a measure of the retention of liquids in the absorbent materials.

The absorbent material is placed within a "teabag", immersed in a 0.9% by weight sodium chloride solution for 20 minutes, and then centrifuged for 3 minutes. The ratio of the retained liquid weight to the initial weight of the dry material is the absorptive capacity of the absorbent material.

Two litres of 0.9% by weight sodium chloride in distilled water is poured into a tray having dimensions 24 cm x 30 cm x 5 cm. The liquid filling height should be about 3 cm.

The teabag pouch has dimensions 6.5 cm x 6.5 cm and is available from Teekanne in Düsseldorf, Germany. The pouch is heat sealable with a standard kitchen plastic bag sealing device (e.g. VACUPACK2 PLUS from Krups, Germany).

The teabag is opened by carefully cutting it partially, and is then weighed. About 0.200g of the sample of the absorbent material, accurately weighed to  $\pm 0.005$ g, is placed in the teabag. The teabag is then closed with a heat sealer. This is called the sample teabag. An empty teabag is sealed and used as a blank.

The sample teabag and the blank teabag are then laid on the surface of the saline solution, and submerged for about 5 seconds using a spatula to allow complete wetting (the teabags will float on the surface of the saline solution but are then completely wetted). The timer is started immediately.

After 20 minutes soaking time the sample teabag and the blank teabag are removed from the saline solution, and placed in a Bauknecht WS130, Bosch 772 NZK096 or equivalent centrifuge (230 mm diameter), so that each bag sticks to the outer wall of the centrifuge basket. The centrifuge lid is closed, the centrifuge is started, and the speed increased quickly to 1,400 rpm. Once the centrifuge has been stabilised at 1,400 rpm the timer is started. After 3 minutes, the centrifuge is stopped.

The sample teabag and the blank teabag are removed and weighed separately.

The Teabag Centrifuge Capacity (TCC) for the sample of absorbent material is calculated as follows:

$$\text{TCC} = [(\text{sample teabag weight after centrifuging}) - (\text{blank teabag weight after centrifuging}) - (\text{dry absorbent material weight})] \div (\text{dry absorbent material weight}).$$

Also, specific parts of the structures or the total absorbent articles can be measured, such as "sectional" cut outs, i.e. looking at parts of the structure or the total article, whereby the cutting is done across the full width of the article at

determined points of the longitudinal axis of the article. In particular, the definition of the "crotch region" as described above allows to determine the "crotch region capacity". Other cut-outs can be used to determine a "basis capacity" (i.e. the amount of capacity contained in a unit area of the specific region of the article. Depending on the size of the unit area (preferably 2 cm by 2 cm) the defines how much averaging is taking place - naturally, the smaller the size, the less averaging will occur.

#### Ultimate Storage Capacity

In order to determine or evaluate the Ultimate Design Storage Capacity of an absorbent article, a number of methods have been proposed.

In the context of the present invention, it is assumed, that the Ultimate Storage Capacity of an article is the sum of the ultimate absorbent capacities of the individual elements or material. For these individual components, various well established techniques can be applied as long as these are applied consistently throughout the comparison. For example, the Tea Bag Centrifuge Capacity as developed and well established for superabsorbent polymers (SAP) can be used for such SAP materials, but also for others (see above).

Once the capacities for the individual materials are known, the total article capacity can be calculated by multiplying these values (in ml/g) with the weight of the material used in the article.

For materials having a dedicated functionality other than ultimate storage of fluids - such as acquisition layers and the like - the ultimate storage capacity can be neglected, either as such materials do in fact have only very low capacity values compared to the dedicated ultimate fluid storage materials, or as such materials are intended to not be loaded with fluid, and thus should release their fluid to the other ultimate storage materials.

#### Density/caliper/basis weight measurement

A specimen of a defined area such as by cutting with a sample cutter is weighed to at least 0.1% accuracy. Caliper is measured under an applied pressure of 550 Pa (0.08 psi) for an test area of 50 mm diameter. Basis weight as weight per unit area expressed in g/m<sup>2</sup>, caliper expressed in mm @ 550 Pa pressure, and density expressed in g/cm<sup>3</sup> can be readily calculated.

**Claims**

1. Fluid handling member having a Capillary Sorption Desorption Height at 50% of the capacity at 0 cm height (CSDH 50); further having a liquid permeability at 100% saturation  $k(100)$  and at 50% saturation ( $k(50)$ ), characterized in that the CSDH 50 value is less than about 150 cm, and in that  $k(100)$  has a value of more than about 1 Darcy, and in that  $k(50)$  has a value, which is more than about 14% of  $k(100)$ .
2. Fluid handling member according to claim 1, wherein the member has a  $k(100)$  value of more than about 2 Darcy.
3. Fluid handling member according to claim 1, wherein the member has a  $k(100)$  value of more than about 8 Darcy.
4. Fluid handling member according to claim 1, wherein the member has a  $k(100)$  value of more than about 100 Darcy.
5. Fluid handling member according to any of claims 1 to 4, wherein the member has a  $k(50)$  value of more than about 18 % of  $k(100)$ .
6. Fluid handling member according to claim 5, wherein the member has a  $k(50)$  value of more than about 25 % of  $k(100)$ .
7. Fluid handling member according to claim 6, wherein the member has a  $k(50)$  value of more than about 35 % of  $k(100)$ .
8. Fluid handling member according to any of claims 1 to 7, wherein the member has a permeability at 30 % of its saturation  $k(30)$  which is more than about 3.5% of  $k(100)$ .
9. Fluid handling member according to claim 8, wherein the member has a  $k(30)$  value, which is more than about 5% of  $k(100)$ .
10. Fluid handling member according to claim 9, wherein the member has a  $k(30)$  value, which is more than about 10% of  $k(100)$ .

11. Fluid handling member according to any of claims 1 to 10, wherein the member has a CSDH 50 value of less than about 100 cm.
12. Fluid handling member according to claim 11, wherein the member has a CSDH 50 value of less than about 75 cm.
13. Fluid handling member according to claim 12, wherein the member has a CSDH 50 value of less than about 50 cm.
14. Fluid handling member having a Capillary Sorption Desorption Height (CSDH 50) further having a liquid permeability at 100% saturation  $k(100)$  and at 50% saturation ( $k(50)$ ), characterized in that it has a CSDH 50 value of less than about 150 cm, and in that it has a permeability  $k(100)$  at 100% saturation has a value of more than about 1 Darcy, and in that it has a permeability at 50% of its saturation  $k(50)$  which is more than about 14% of  $k(100)$ , and in that it has a flux at 15 cm wicking height of at least  $0.045 \text{ g/cm}^2/\text{sec}$  in the Vertical Wicking Test as described herein.
15. A fluid handling member according to claim 14, having a flux at 15 cm wicking height of at least  $0.06 \text{ g/cm}^2/\text{sec}$ .
16. A fluid handling member according to claim 14, having a flux at 15 cm wicking height of at least  $0.1 \text{ g/cm}^2/\text{sec}$ .
17. Fluid handling member according to claim 14, wherein the member has a  $k(100)$  value of more than about 2 Darcy.
18. Fluid handling member according to claim 14, wherein the member has a  $k(100)$  value of more than about 8 Darcy.
19. Fluid handling member according to claim 14, wherein the member has a  $k(100)$  value of more than about 100 Darcy.

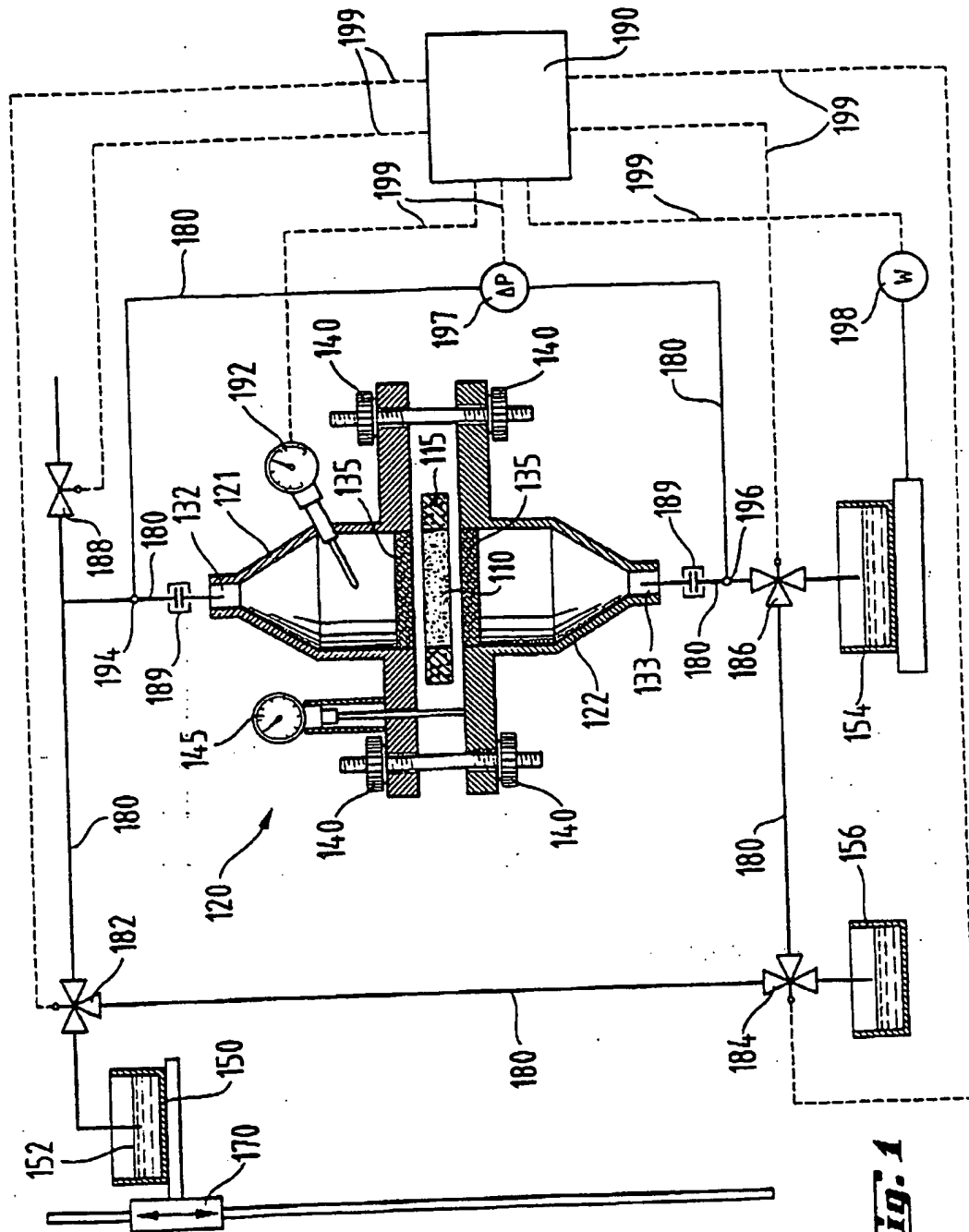
20. Fluid handling member according to any of claims 14 - 19, wherein the member has a  $k(50)$  value of more than about 18 % of  $k(100)$ .
21. Fluid handling member according to claim 20, wherein the member has a  $k(50)$  value of more than about 25 % of  $k(100)$ .
22. Fluid handling member according to claim 20, wherein the member has a  $k(50)$  value of more than about 35 % of  $k(100)$ .
23. Fluid handling member according to any of claims 14 - 22, wherein the member has a permeability at 30 % of its saturation  $k(30)$  which is more than about 3.5% of  $k(100)$ .
24. Fluid handling member according to claim 23 wherein the member has a  $k(30)$  value, which is more than about 5% of  $k(100)$ .
25. Fluid handling member according to claim 23 wherein the member has a  $k(30)$  value, which is more than about 10% of  $k(100)$ .
26. Fluid handling member according to any of claims 14 - 25, wherein the member has a CSDH 50 value of less than about 100 cm.
27. Fluid handling member according to any of claims 14 - 26, wherein the member has a CSDH 50 value of less than about 75 cm.
28. Fluid handling member according to any of claims 14 - 27, wherein the member has a CSDH 50 value of less than about 50 cm.
29. Fluid handling member according to any of the preceding claims, further characterized in that it comprises a hydrophilic, flexible polymeric foam structure of interconnected open-cells.
30. Fluid handling member according to claim 29, further characterized in that it expands upon wetting..
31. Fluid handling member according to claim 29, whereby it re-collapses upon loosing liquid.

32. Fluid handling member according to any of claims 29 to 31 having a capillary collapse pressure of at least about 15 cm.
33. Fluid handling member according to any of claims 29 to 32 having a free absorbent capacity of at least about 15 g/g.
34. Absorbent article comprising a Fluid handling member according to any of the preceding claims, additionally comprising a first liquid storage region-fluid communication with said Fluid Handling Member.
35. Absorbent article according claim 34, further comprising at least a second liquid storage region, whereby both said liquid storage regions are in liquid communication with said Fluid handling member.
36. Absorbent article according to any of claims claim 34 or 35, wherein at least one of said liquid storage regions comprises material exhibiting a Capillary Sorption Absorption Height at 50% of its maximum capacity (CSAH 50) of at least about 40 cm.
37. An absorbent article according to any of claims 34,  
further characterized in that  
it comprises an ultimate liquid storage material providing at least 80 % of the total ultimate storage capacity of the absorbent core.
38. An absorbent article according to any of claims 34,  
further characterized in that  
it comprises a ultimate liquid storage material providing at least 90 % of the total ultimate storage capacity of the absorbent core.
39. Absorbent article comprising an absorbent core comprising a crotch region and one or more waist regions, whereby said crotch region has a lower ultimate fluid storage capability than said one or more waist regions together, and wherein said crotch region comprises a Fluid handling member according to any of claims 1 to 33.

40. An absorbent article according to claim 39, wherein said crotch region has an ultimate fluid storage basis capacity of less than 0.9 times the average ultimate fluid storage basis capacity of the absorbent core.
41. An absorbent article according to claim 40, wherein said crotch region has an ultimate fluid storage basis capacity of less than 0.5 times the average ultimate fluid storage basis capacity of the absorbent core.
42. An absorbent article according to claim 41, wherein said crotch region has an ultimate fluid storage basis capacity of less than 0.3 times the average ultimate fluid storage basis capacity of the absorbent core.
43. An absorbent article according to claim 39 wherein said crotch region has a sectional ultimate fluid storage capacity of less than 49% of the total core ultimate fluid storage capacity.
44. An absorbent article according to claim 39 wherein said crotch region has a sectional ultimate fluid storage capacity of less than 41% of the total core ultimate fluid storage capacity.
45. An absorbent article according of claim 39 wherein said crotch region has a sectional ultimate fluid storage capacity of less than 23 % of the total core ultimate fluid storage capacity.
46. An absorbent article according to any of claims 39 to 45, further characterised in that at least 50% of the area of said crotch region contain essentially no ultimate storage capacity.
47. An absorbent article according to any of claims 39 to 46, further characterised in that less than 50% of said ultimate storage capacity are positioned forwardly from the crotch zone in the front half of the article, and more than 50% of said ultimate storage capacity are positioned in the rear half of the article.
48. An absorbent article according to claim 47, wherein less than 33% of said ultimate storage capacity are positioned forwardly from the crotch zone / in



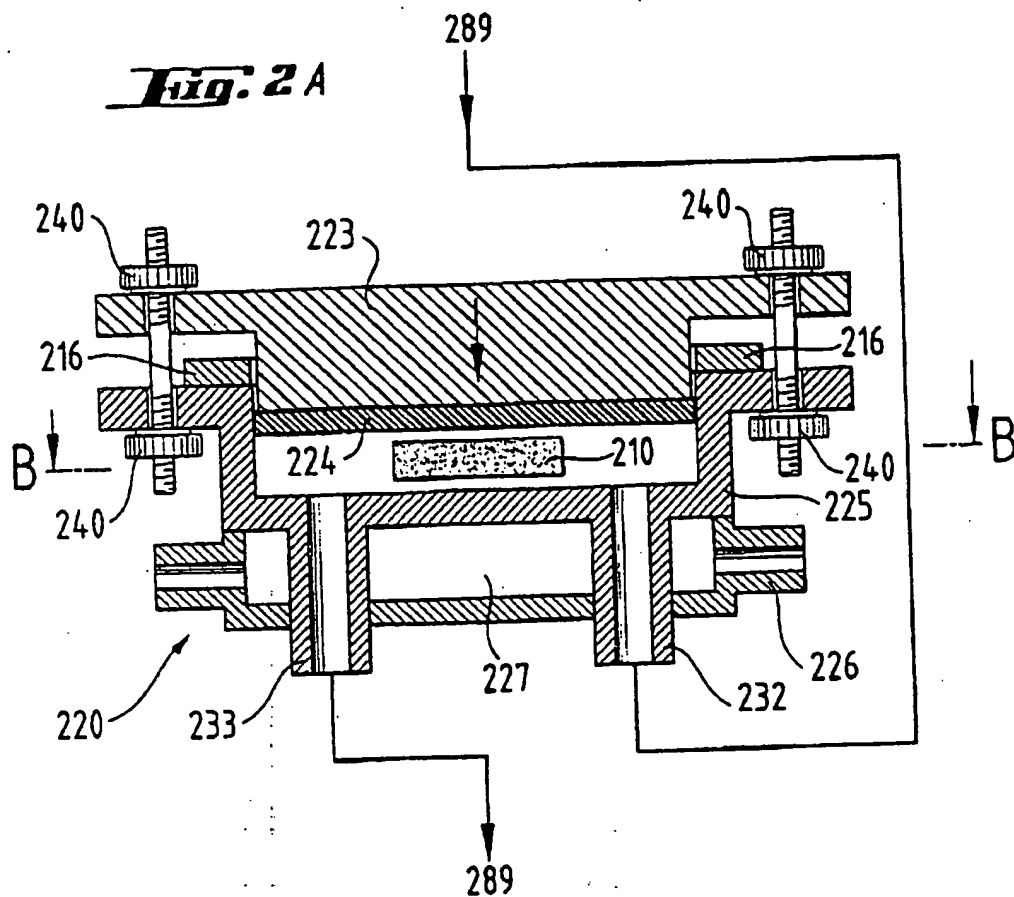
the front half of the article, and more than 67% of said ultimate storage capacity are positioned in the rear half of the article.



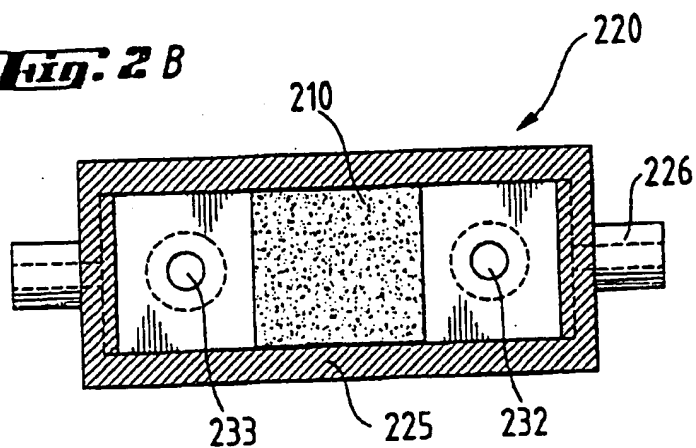
**Fig. 1**

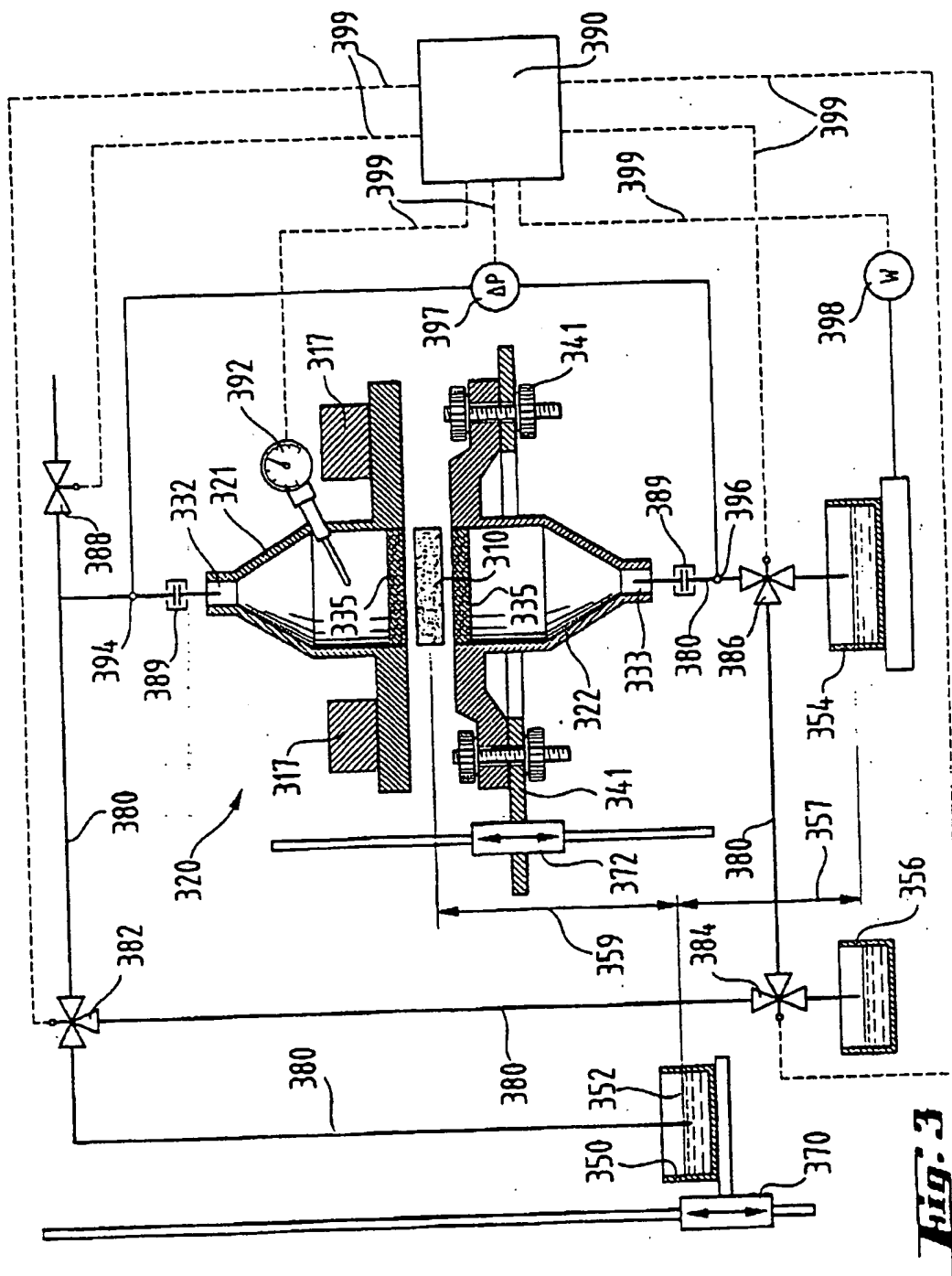
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**Fig. 2 A**



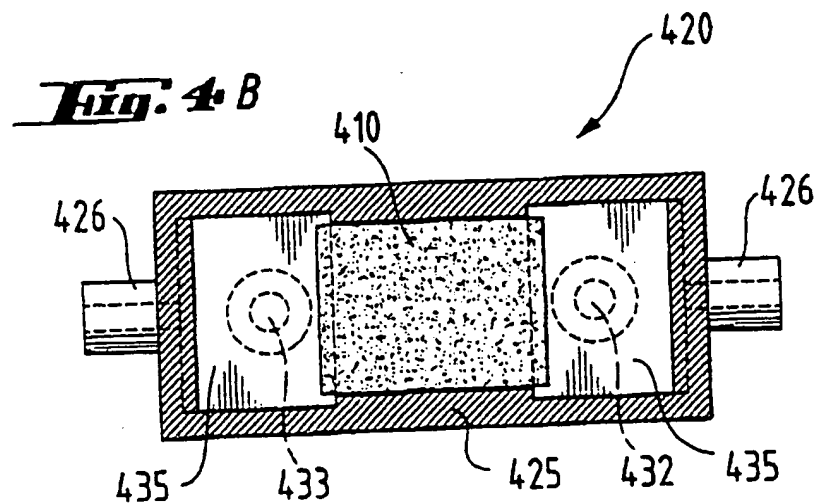
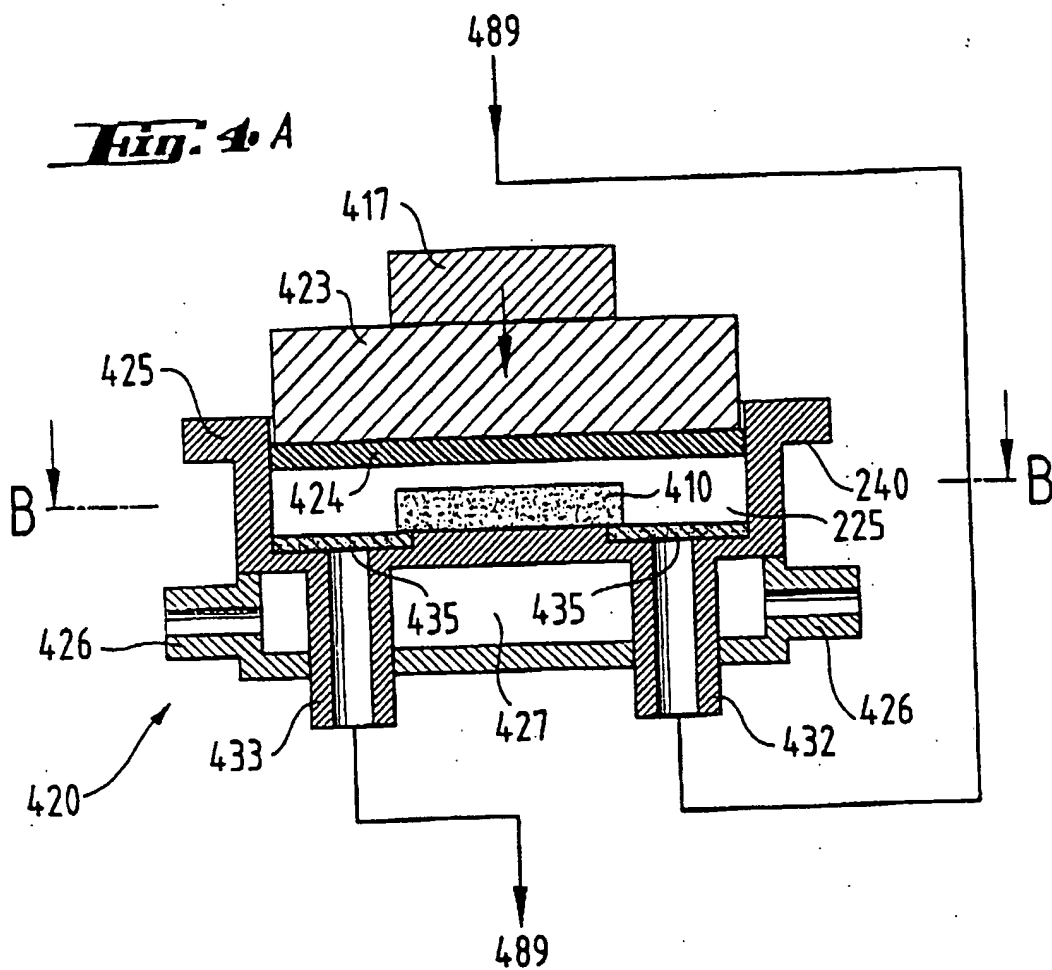
**Fig. 2 B**



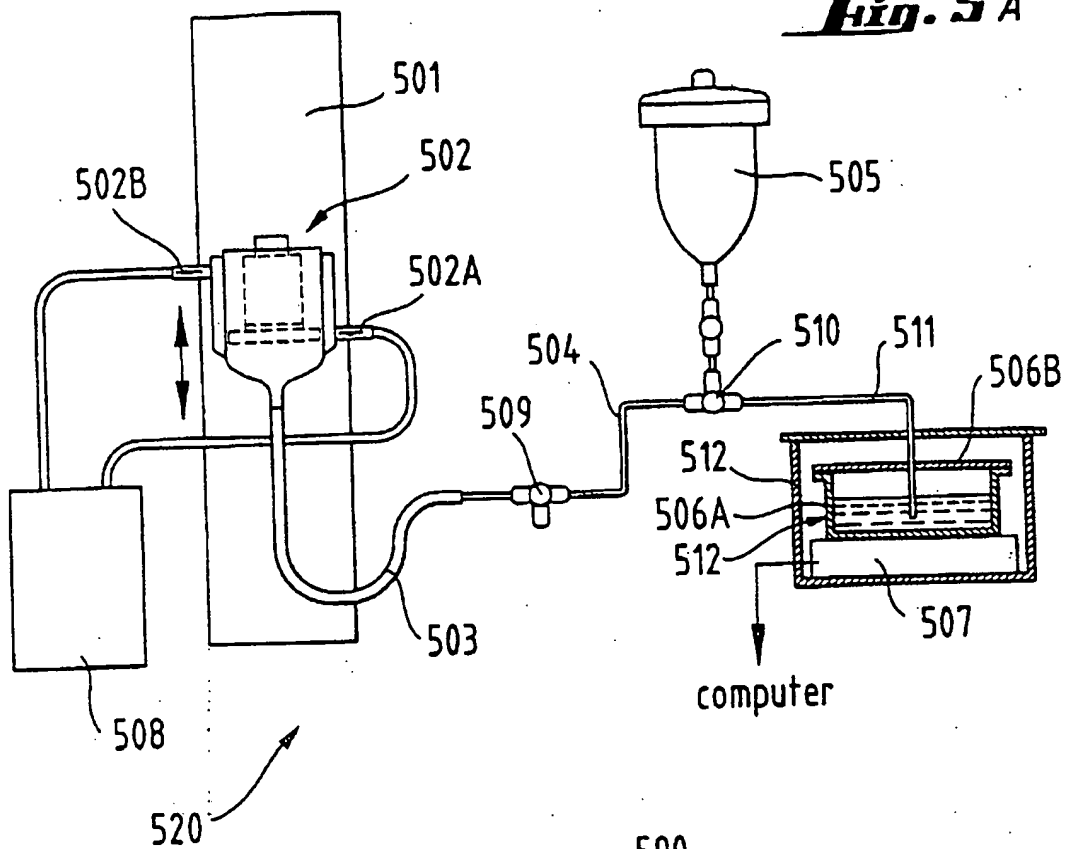
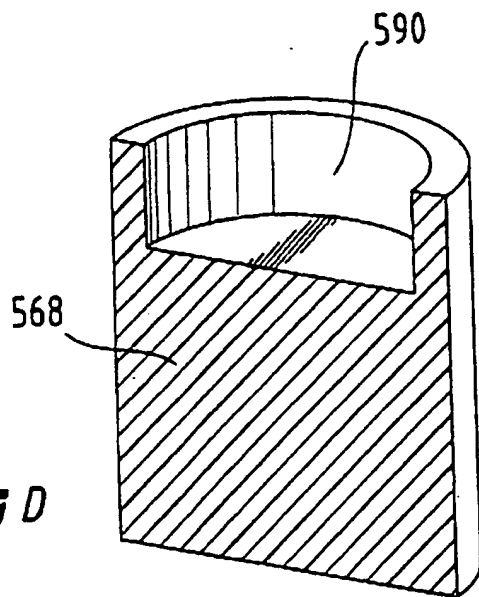


**Fig. 3**

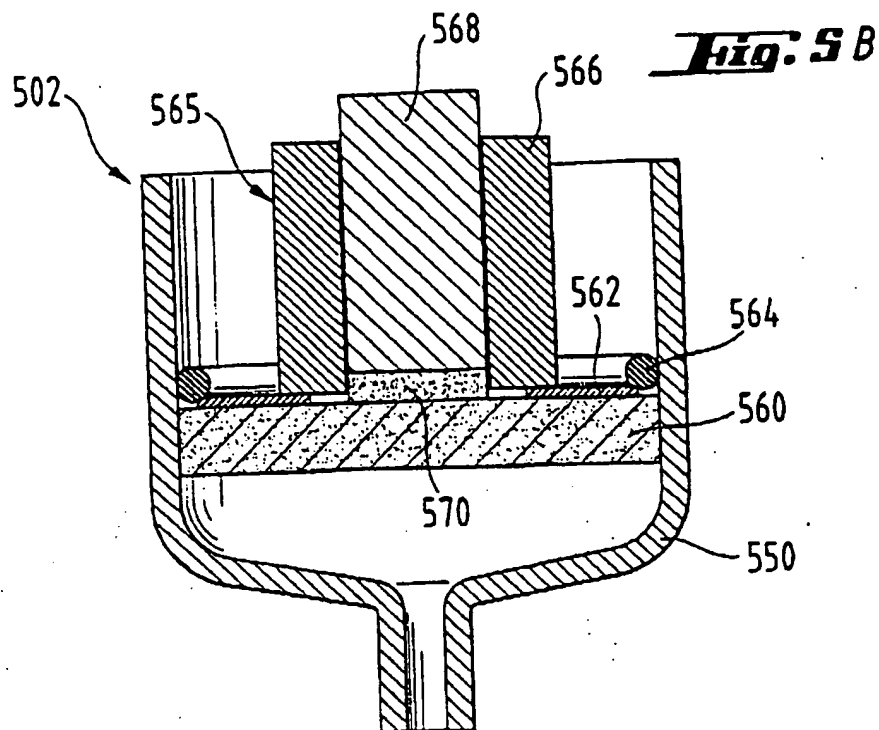
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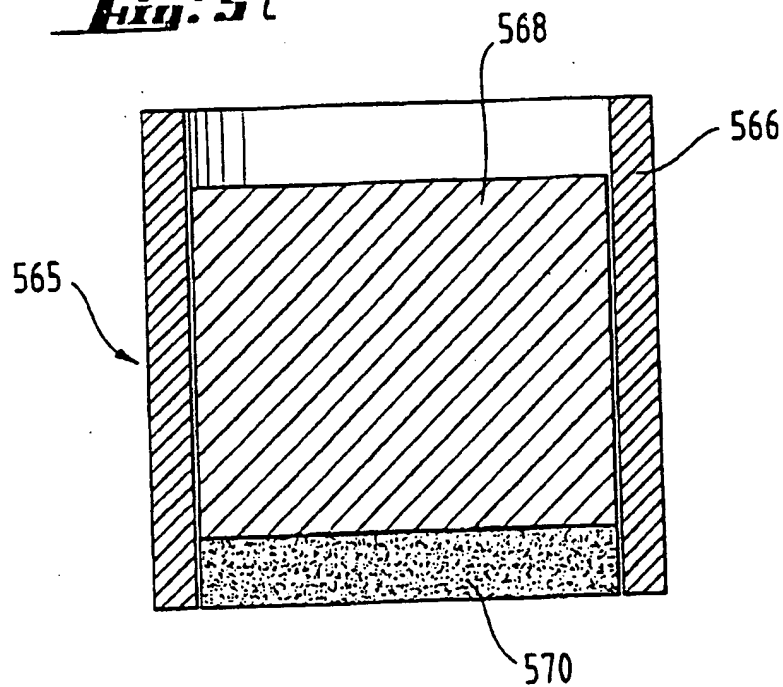
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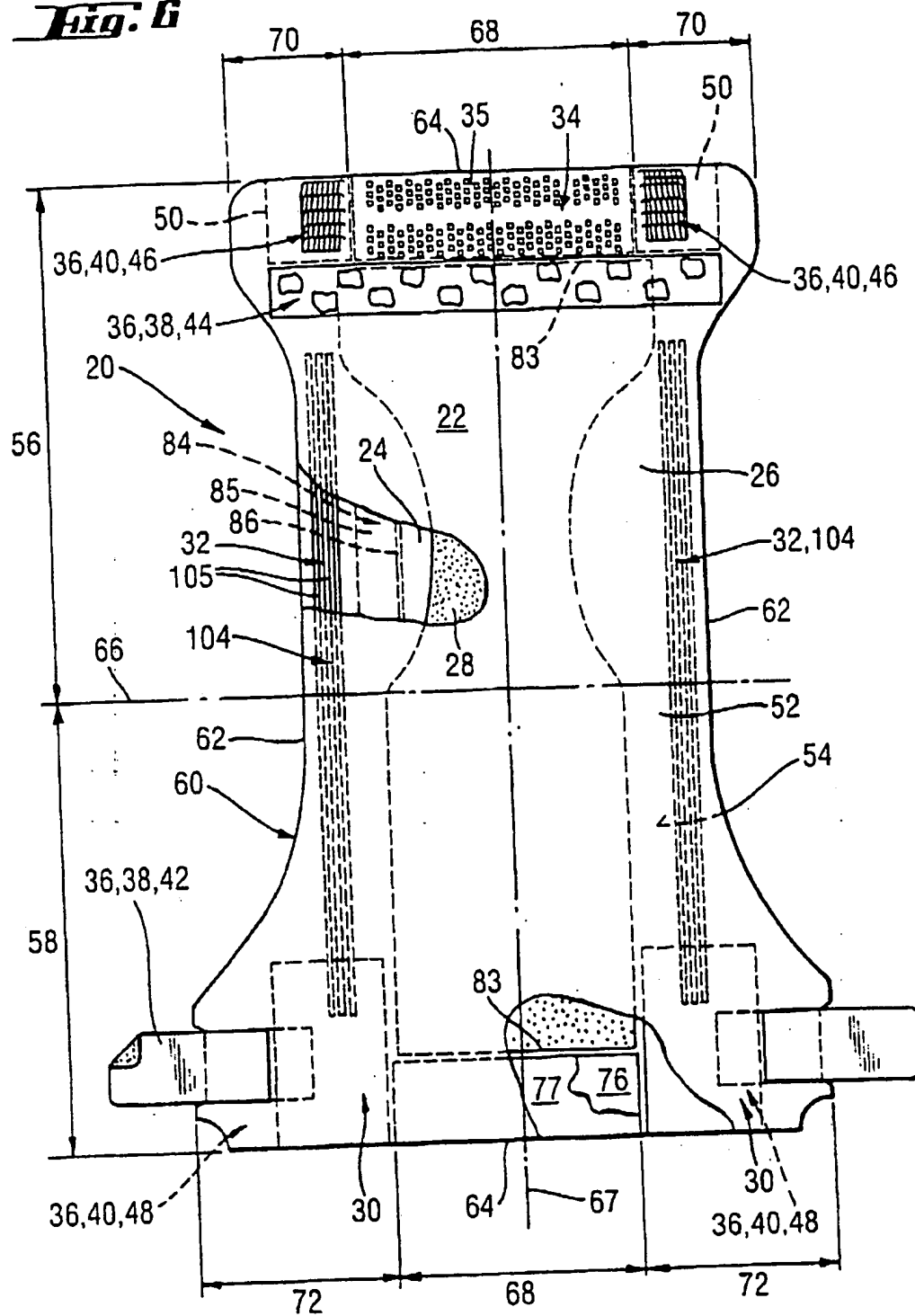
**Fig. 5 A****Fig. 5 D**

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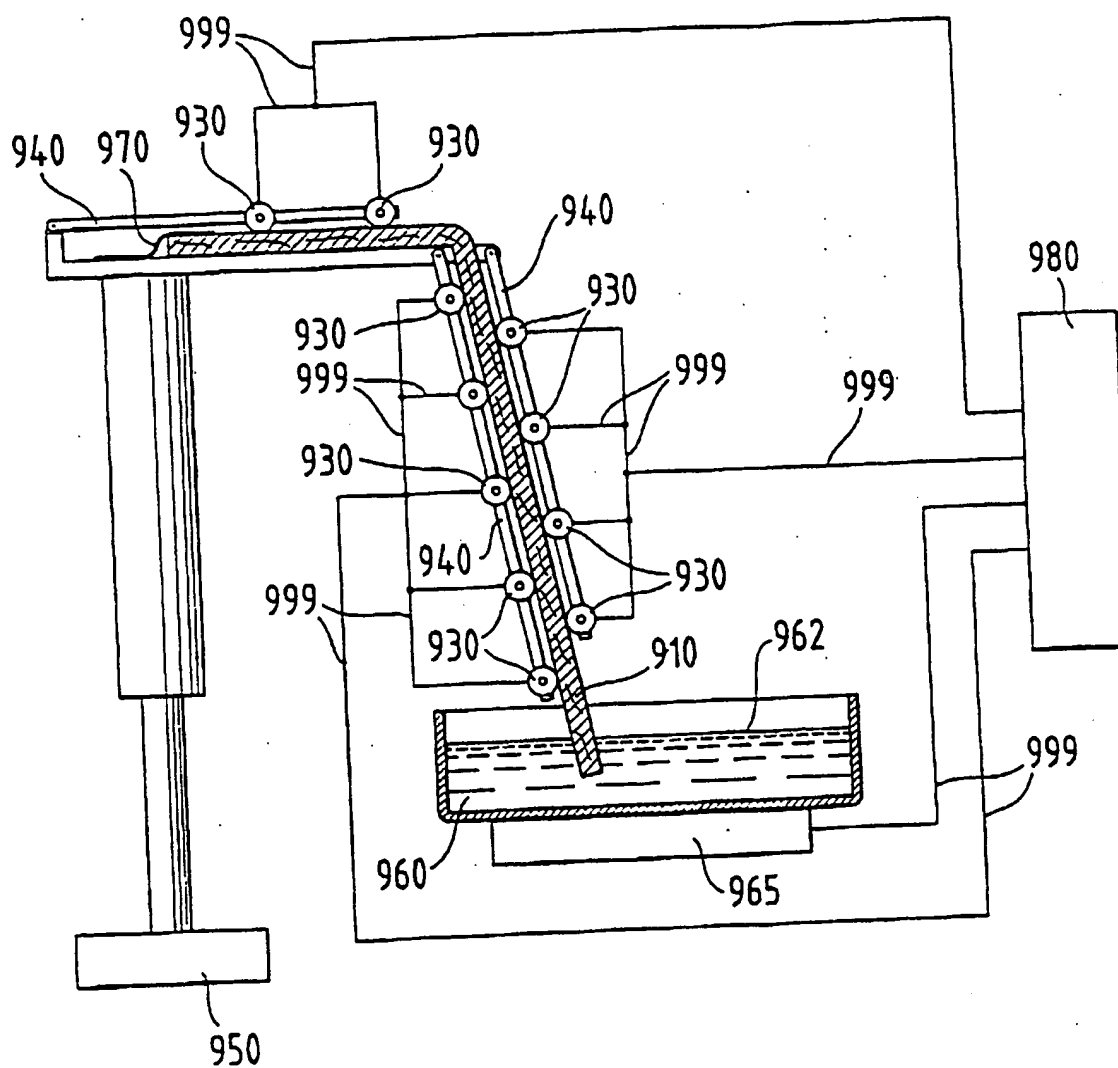


**Fig. 5C**



**Fig. 6**



**Fig. 7**

# INTERNATIONAL SEARCH REPORT

Intern. Application No

PCT/IB 99/00409

## A. CLASSIFICATION OF SUBJECT MATTER

IPC 6 A61F13/15

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 A61F A61L

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 5 147 345 A (YOUNG GERALD A ET AL) 15 September 1992  see column 3, line 48 - line 52; claims; figures 3-6	1,29-31, 34,35, 37-48
A	GB 2 296 511 A (KIMBERLY CLARK CO) 3 July 1996 see claims 1,12-15,20,21	1-14, 17-25
A	WO 97 34558 A (PROCTER & GAMBLE) 25 September 1997 see claims; figures	1,30-36
A	EP 0 809 991 A (PROCTER & GAMBLE) 3 December 1997 cited in the application see claims	1,14-16

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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Date of the actual completion of the international search

27 May 1999

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Mirza, A

# INTERNATIONAL SEARCH REPORT

International Application No

PCT/IB 99/00409

## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	<p>W0 97 23184 A (KIMBERLY CLARK CO)</p> <p>3 July 1997</p> <p>see abstract</p> <p>-----</p>	1,14-16

# INTERNATIONAL SEARCH REPORT

Information on patent family members

Intern. Appl. Application No

PCT/IB 99/00409

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 5147345 A	15-09-1992	AT 149824 T AU 669667 B AU 2460892 A BR 9206373 A CA 2114957 A CN 1074110 A CZ 9400303 A DE 69218219 D DE 69218219 T DK 598823 T EG 19687 A EP 0598823 A ES 2099831 T FI 940650 A GR 3022800 T HK 1006530 A HU 71533 A JP 6509729 T MX 9204675 A NO 940451 A NZ 243904 A PL 169450 B PT 8582 U PT 101751 A,B SG 48921 A SK 18194 A TR 26574 A WO 9303699 A US 5652194 A US 5649920 A US 5318554 A US 5387207 A	15-03-1997 20-06-1996 16-03-1993 25-04-1995 04-03-1993 14-07-1993 13-07-1994 17-04-1997 10-07-1997 21-04-1997 31-10-1995 01-06-1994 01-06-1997 11-02-1994 30-06-1997 05-03-1999 28-12-1995 02-11-1994 01-03-1993 11-04-1994 26-09-1995 31-07-1996 31-05-1993 31-01-1996 18-05-1998 07-12-1994 15-03-1995 04-03-1993 29-07-1997 22-07-1997 07-06-1994 07-02-1995
GB 2296511 A	03-07-1996	US 5849000 A AU 4527096 A CA 2209152 A CN 1175215 A EP 0800410 A FR 2729298 A HU 77842 A JP 10511593 T PL 321788 A SK 86197 A WO 9620739 A ZA 9510976 A	15-12-1998 24-07-1996 11-07-1996 04-03-1998 15-10-1997 19-07-1996 28-08-1998 10-11-1998 22-12-1997 04-03-1998 11-07-1996 25-06-1996
WO 9734558 A	25-09-1997	US 5855572 A AU 2341897 A CA 2249296 A EP 0891176 A	05-01-1999 10-10-1997 25-09-1997 20-01-1999
EP 0809991 A	03-12-1997	AU 3065097 A AU 3147097 A WO 9745085 A WO 9745087 A	05-01-1998 05-01-1998 04-12-1997 04-12-1997
WO 9723184 A	03-07-1997	US 5843852 A AU 1337597 A	01-12-1998 17-07-1997

# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/IB 99/00409

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
WO 9723184 A		CA 2239169 A	03-07-1997
		EP 0876132 A	11-11-1998
		AU 5511098 A	15-07-1998
		WO 9826742 A	25-06-1998
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